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VOLUME 3
FINAL QUALITY ASSURANCE PROJECT PLAN
HIMCO DUMP
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
ELKHART, INDIANA
JUNE 1990

Prepared for:

U.S. Environmental Protection Agency
Emergency and Remedial Response Branch
Region V
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EPA CONTRACT NO: 68-W8-0093
WORK ASSIGNMENT NO: 17-514J
DONOHUE & ASSOCIATES, INC.

VOLUME 3
QUALITY ASSURANCE PROJECT PLAN
HIMCO DUMP RI/FS
ELKHART, INDIANA

JUNE 1990

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A SAS Request Forms:

Water Quality SAS:

Chloride, sulfate, TDS, TSS,
alkalinity, TP, TKN, NH₃, NO₂ and
NO₃, COD

LIST OF APPENDICES
(Continued)

Bromide SAS:	Anions, ion-exchange chromatographic, automated
Residential Wells Organic SAS:	Analysis of drinking water/residential wells for volatiles, semi-volatiles, and pesticides/PCBs with low quantitation limits
Residential Wells Inorganic SAS:	Analysis of drinking water/residential wells for metals and cyanide
Sorbent Tube SAS:	Analysis of sorbent tubes for TCL volatile organics
Geotech SAS:	Analysis of soil samples for Atterburg Limits, particle size analysis, permeability, consolidation, and triaxial shear
Organic Carbon SAS:	Determination of percent organic carbon in soil on air dried sample
B	DQO Summary Sheets
C	Sample Bottle Cleaning Protocols
D	Field Meter Calibration Procedures
E	Standard Operating Procedures for Field Measurements

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ACRONYMS

Himco Dump QAPP
Elkhart, Indiana

ABN	Acid and Base-Neutral Semivolatile Organic Compounds
ASTM	American Society of Testing Materials
BNA	Base-Neutral and Acid Semivolatile Organic Compounds (same as ABN)
BOD	Biochemical Oxygen Demand
CDO	Central District Office, U.S. EPA Region V
CH ₄	Methane
Cl	Chloride
CLP	Contract Laboratory Program
CN	Cyanide
COD	Chemical Oxygen Demand
COE	Corps of Engineers, U.S. Army
CRDL	Contract Required Detection Limit
CRL	Central Regional Laboratory, U.S. EPA Region V
CRQL	Contract Required Quantitation Limit
DO	Dissolved Oxygen
DQO	Data Quality Objective
E&E	Ecology & Environment, Inc.
EM	Electromagnetic Meter
EMSL	Environmental Monitoring and Support Laboratory, U.S. EPA
EPA	U.S. Environmental Protection Agency
FIT	Field Investigation Team
FS	Feasibility Study
FSP	Field Sampling Plan
FTL	Field Team Leader, Donohue
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption
HNO ₃	Nitric Acid
HNu	Photoionization detector manufacturer
HQ	Headquarters, U.S. EPA
H ₂ S	Hydrogen Sulfide
IADS	Inorganic Analysis Data Sheet
ICP	Inductively Coupled Argon Plasma Spectrometer
I.D.	Inner Diameter
IDEM	Indiana Department of Environmental Management
IDL	Instrument Detection Limit
ISBH	Indiana State Board of Health
LSSS	Laboratory Support Services Section, U.S. EPA Region V
Lumidor	Gas monitoring device manufacturer
MCL	Maximum Contaminant Level
NC	Not Calculated
NEIC	National Enforcement Investigations Center, U.S. EPA
NH ₃	Ammonia Nitrogen
NO ₂ +NO ₃	Nitrite + Nitrate Nitrogen

ACRONYMS

Himco Dump QAPP
Elkhart, Indiana

NPL	National Priorities List
OADS	Organic Analysis Data Sheet
OERR	Office of Emergency and Remedial Response, U.S. EPA
PCBs	Polychlorinated Biphenyls
PCB/P	PCBs and Pesticides
pH	Measure of acidity indicated as log of hydrogen ion concentration
PM	Project Manager, Donohue
PRP	Potentially Responsible Party
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAMS	Quality Assurance Management Staff, U.S. EPA
QAPP	Quality Assurance Project Plan
QC	Quality Control
%R	Percent Recovery
RAS	Routine Analytical Services
RI	Remedial Investigation
RMCL	Recommended Maximum Contaminant Level
RPD	Relative Percent Difference
RPM	Remedial Project Manager, U.S. EPA
RPO	Remedial Project Officer, U.S. EPA
RSCC	Regional Sample Control Center, U.S. EPA
SAS	Special Analytical Services
SM	Site Manager, Donohue
SMO	Sample Management Office
SO ₄	Sulfate
SOP	Standard Operating Procedure
SOW	Statement of Work
SQCO	Site Quality Control Officer, Donohue
TAC	Technical Advisory Committee, Donohue
TAL	Target Analyte List
TCL	Target Compound List
TDS	Total Dissolved Solids
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TP	Total Phosphorus
TSQAM	Technical Services Quality Assurance Manager, Donohue
TSS	Total Suspended Solids
USGS	United States Geological Survey
VOA	Volatile Organic Compounds
VOC	Volatile Organic Compounds (same as VOA)

3.0 PROJECT DESCRIPTION

3.1 INTRODUCTION

Donohue & Associates, Inc., (Donohue) is submitting this Quality Assurance Project Plan (QAPP) for the Himco Dump Superfund Site RI/FS to the U.S. Environmental Protection Agency (EPA), Region V, in response to EPA Work Assignment No. 17-5L4J (EPA Contract No. 68-W8-0093). This document is Volume 3 of the Himco Dump Work Plan and related project plans prepared by Donohue. The purpose of the RI is to determine the nature, extent, and sources of contamination at the Himco Dump site, and to gather data necessary to support a baseline risk assessment and a feasibility study. The investigation will include waste characterization, determination of the presence/absence of wetlands, surface water, sediment, and soil sampling analysis, waste mass gas investigation, determination of hydrogeologic characteristics, and groundwater characterization.

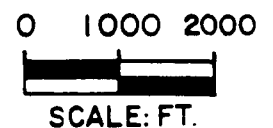
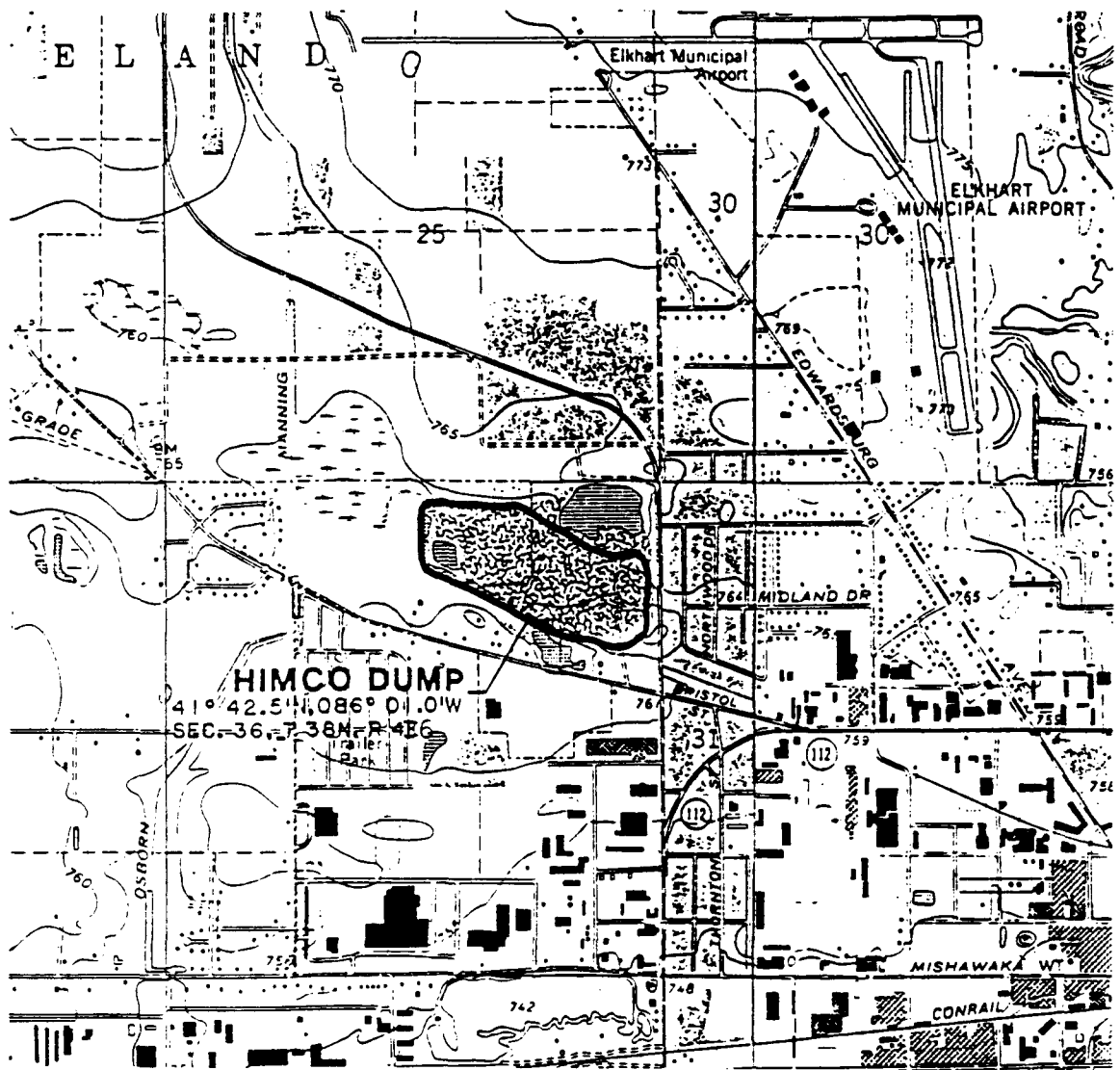
The purpose of the FS is to develop and evaluate appropriate remedial action alternatives based on technical, environmental, public health, and economic considerations, and to determine the most applicable remedial action alternative for the site.

The purpose of the Quality Assurance Project Plan (QAPP) is to present the planned system of activities and expected level of data quality for the RI/FS. The precision, accuracy, completeness, representativeness, and comparability of the environmental data collected must be known and documented according to this plan. Presented in this QAPP are the personnel responsible for quality assurance, the data quality objectives, and the specific quality control measures to be taken to reach the objectives.

This QAPP has been prepared in accordance with the 16 QAPP elements as specified in EPA QAMS 005/80, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," and the "Region V Content Requirements for QAPP" draft prepared by Cheng-Wen Tsai, February 1987, revised January 1989. Preparation of this QAPP was accomplished after meetings on project scope, data quality objectives, and QAPP contents were held between EPA, Indiana Department of Environmental Management (IDEM), and Donohue.

3.2 SITE DESCRIPTION

The Himco Dump Site is a closed and covered landfill located at County Road 10 and the Nappanee Street extension in the town of Elkhart, located in Elkhart County, Indiana (Figure 3-1). The site covers approximately 50 acres in the Northeast quarter of Section 36, Township 38 North, Range 4 East, in Cleveland Township. The site is bounded on the north by a tree line and northernmost extent of the gravel pit pond; on the west by the fish pond; on the south by



SOURCE: USGS 7.5 MIN. QUAD ELKHART, INDIANA, 1961
PHOTOREVISED 1981

Donohue

20026

SITE LOCATION MAP

JANUARY, 1990

Engineers • Architects • Scientists

HIMCO DUMP SITE
ELKHART COUNTY, INDIANA



FIGURE 3 -1

County Road 10 and the private residences; and on the east by the Nappanee Street extension. The vicinity of the site is agricultural, residential, and light industrial. Site features and existing monitoring wells are shown in Figure 3-2.

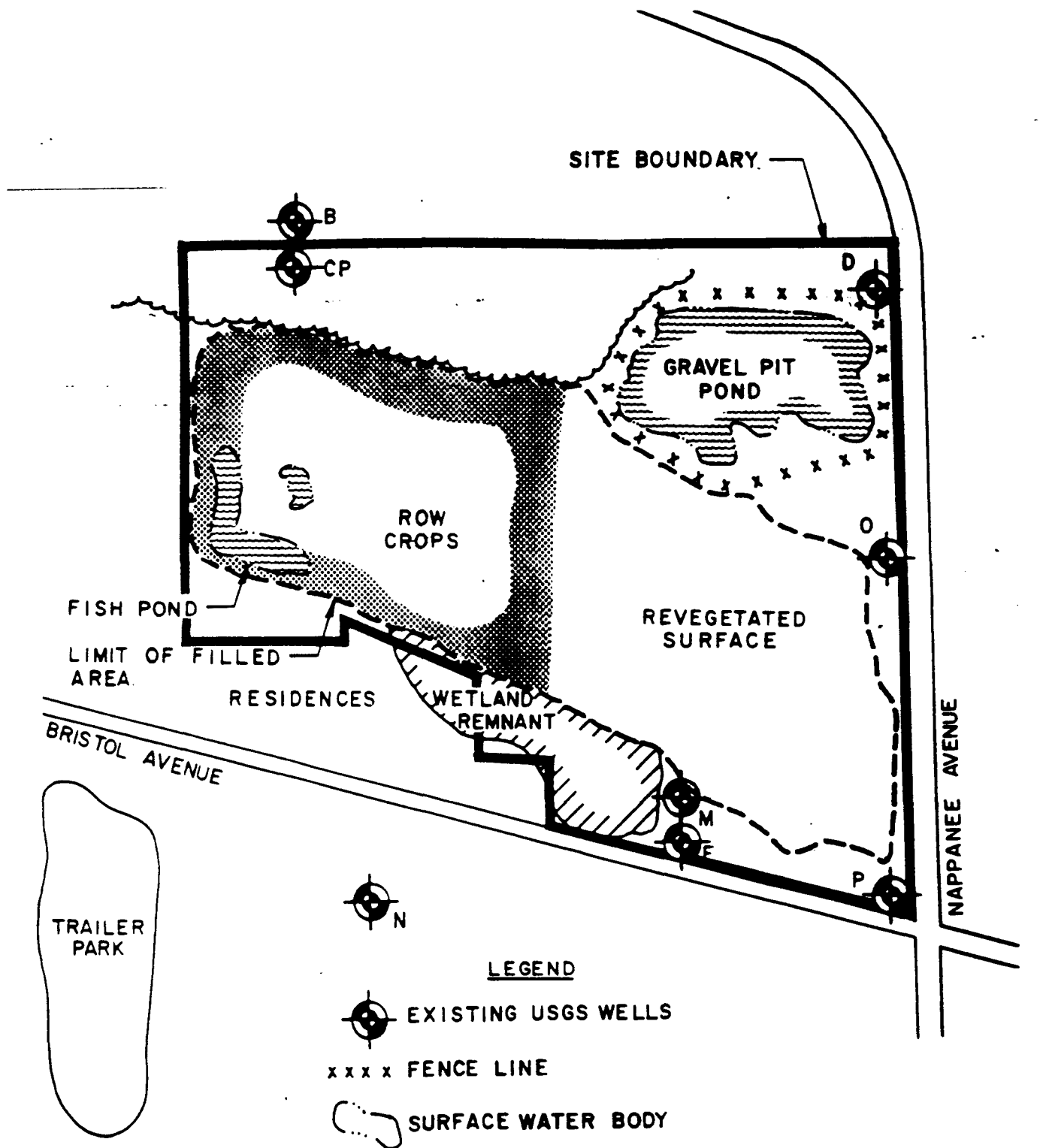
Donohue conducted a site visit on November 9, 1989, to the Himco Dump with representatives of the Indiana Department of Environmental Management (IDEM) and the potentially responsible parties (PRPs). The purpose of the site visit was to observe obvious surficial areas of environmental concern (e.g. stressed vegetation, stained soils, and uncontrolled dumping), determine site access points for field operations, and inspect the final cover for uncovered refuse and surface water drainage patterns.

During the site visit, the final cover of sand overlying calcium sulfate was observed. The thickness of sand across the site was observed to be 0 to 1 foot thick. The thickness of the calcium sulfate layer is unknown. The western half of the dump was used as cropland (for soybeans) up to one year ago. The eastern half of the site is covered by grassland with some tree stands. There is an access road (made of sand) into the site near the intersection of County 10 and Nappanee Street. There is an abandoned gravel pit operation in the northeast corner of the site. A truck scale, concrete structures, and a utility pole are in this area. The gravel pit itself is filled with water, and a steep drop-off was observed. Some minor dumping into the gravel pit pond was noted. Another pond exists in the southwest corner of the site. It was reported that the owner of the property excavated this area to create a "fishing hole" which was then stocked with fish. It is not known if any biota still exist in this pond.

Surface water drainage across the Himco Dump site is probably radial due to the configuration of the landfill. The highest point of the landfill is probably near its geographic center at a reported height of 15 feet above ground surface sloping to a height of 0 to 5 feet around the dump's perimeter. Erosional areas were noted around the site, some of them penetrating the calcium sulfate layer. Paper and plastic refuse also lay uncovered in certain areas. There were also deep caverns noted in areas that were initially created by burrowing rodents. Wildlife observed at the site included a four-point buck and various species of birds.

On-site and off-site monitoring wells installed by the USGS were also inspected by Donohue during the site visit. The wells were constructed of 2 -inch and 4-inch internal diameter (ID) polyvinyl chloride (PVC) and had no protective casings. All wells checked had water in them, and these water levels were recorded. These wells were reported to have been constructed in the early 1980s.

An initial walk-through of the site was conducted with atmospheric monitoring equipment (photoionization detector (HNU), landfill gas detector (Lumidor), and geiger counter). Compounds monitored included: volatile organic com-



Donohue

20026

FEBRUARY, 1990

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EXISTING SITE FEATURES

HMC DUMP SITE
ELKHART COUNTY, INDIANA



FIGURE 9-2

pounds, hydrogen sulfide, methane, X-ray and gamma radiation. No readings above background were detected on any of the instruments. However, "landfill gas" smell was detected intermittently across the dump site.

3.3 SITE HISTORY AND BACKGROUND

The Himco Dump site was a privately owned site operated by Himco Waste Away Service, Inc., that was in operation between 1960 and September 1976. A brief history of the Himco Dump site was provided by Chuck Himes, principal landfill operator, during the site visit on November 9, 1989. The area was initially a marsh and grassland. There was no liner, leachate, or gas recovery system constructed for the landfill. Refuse was placed at ground surface across the site with the exception of trench filling in the eastern quarter of the site. A total of 5 trenches 10 to 15 feet deep, a truck width wide and 30 feet long, were excavated in this area. Paper refuse was reportedly dumped in these trenches and burned. The landfill had no borrow source but obtained sandy soil for daily cover from the gravel pit to the north, an excavated pond to the west, and essentially anywhere around the perimeter of the site where sand was available. It was reported that essentially two-thirds of the waste present in the dump was calcium sulfate from Miles Laboratories. As much as 360 tons/day were dumped over an unknown time duration. Other wastes accepted at the landfill included demolition/construction debris, industrial and hospital wastes, and, to a minor degree, general household refuse. In 1977, the landfill was closed and covered. The cover was constructed of approximately 1 foot of sand overlying 6 inches of calcium sulfate.

In 1971, the Indiana State Board of Health (ISBH) first identified the Himco site as an open dump. In early 1974, nearby residents of the Himco Dump complained to the ISBH about color, taste, and odor problems with their shallow wells.

Mr. Himes was advised by ISBH to replace six shallow water wells for residences immediately south of the landfill on County Road 10. Analyses of these wells by the state showed high levels of manganese. The old wells were finished at depths of approximately 22 feet, and the new wells were finished at depths ranging from 152 to 172 feet below ground surface.

In 1975, Mr. Himes signed a consent agreement with the ISBH Stream Pollution Control Board to close the dump by September of 1976. The final cover consisted of calcium sulfate overlain by sand.

In 1980, the United States Geological Survey (USGS) conducted a hydrogeological study of northwestern Elkhart County, Indiana. The purpose of the USGS study was to define the hydrogeology in the region and to determine the hydrologic effects of the proposed pumping at the Elkhart Municipal Airport located approximately one mile northeast of the Himco Dump. Data collected on the groundwater regime included the thickness and areal extent of unconsolidated deposits, their hydraulic conductivity, specific yield, transmissivity, and storage coefficients. The general groundwater flow patterns and stream-

aquifer connections were also defined (USGS, 1981). The USGS report determined the horizontal and vertical extent of the leachate plume from the Himco Dump using concentrations of bromide in the groundwater. The bromide concentrations in groundwater have been monitored from 1979 until present.

In 1984, the EPA directed a field investigation team (FIT) consisting of their subcontractor, Ecology and Environment (E&E). A site inspection was conducted by E&E at the Himco Dump. Laboratory analysis of wells sampled by the FIT members showed that the groundwater was impacted by metals and semi-volatile and volatile organic compounds. The metals detected included aluminum, arsenic, barium, chromium, cobalt, selenium, beryllium, cadmium, copper, zinc, manganese, lead, nickel, and mercury. The volatile organic compounds (VOCs) detected included: acetone, benzene, phenol, freons, 4-methylphenol, trans 1,2-dichloroethane, 2-butane, chloroethane, and pyrene. At the time of the site inspections, leachate streams were observed by the FIT members.

As of January 1990, the parcels of land which comprise the landfill are owned by the following individuals or corporations:

- I. Miles Laboratories
- II. CLD Corporation
- III. Alonzo Craft, Jr.
- IV. Indiana and Michigan Electric Co.

3.4 CONTAMINANTS OF CONCERN AND POTENTIAL SOURCE AREAS

A site inspection was conducted in July 1984 by Ecology & Environment (E&E). Groundwater, surface water, and sediment samples were taken and analyzed using the Contract Laboratory Program (CLP) for volatile organics, acid/base-neutral organics, polychlorinated biphenyls (PCBs), pesticides, total metals, and cyanide. Results from this study are contained in Table 3-1. Shallow well E/17', immediately downgradient of the site, contained elevated levels of trans 1,2-dichloroethene, pyrene, acenaphthene, 2,4-dinitrotoluene, zinc, cadmium, and aluminum compared to the upgradient well D/19'. Wells M and P which are located on-site nearest the fill contained detectable phenol, 4-methylphenol, acetone, freons, chloroethane, and trans 1,2-dichloroethene. Surface water and sediment quality at downgradient locations did not differ from upgradient locations. The primary suspected contaminant source is the landfill which is known to contain pharmaceutical waste, industrial waste, municipal waste, and calcium sulfate. Primary release mechanisms include particulate and volatile emissions, percolation, runoff, and erosion. These release mechanisms result in a secondary contaminant source which includes soil under and surrounding the landfill. The mechanisms and pathways applicable to the Himco Dump Site are shown in Figure 3-3.

Secondary release mechanisms include percolation which results in potential contamination of groundwater, sediment, surface water, and wetlands. The secondary release mechanisms of runoff and erosion result in contamination of

FIGURE 3-3
CONCEPTUAL SITE MODEL

**Himco Dump
Elkhart, Indiana**

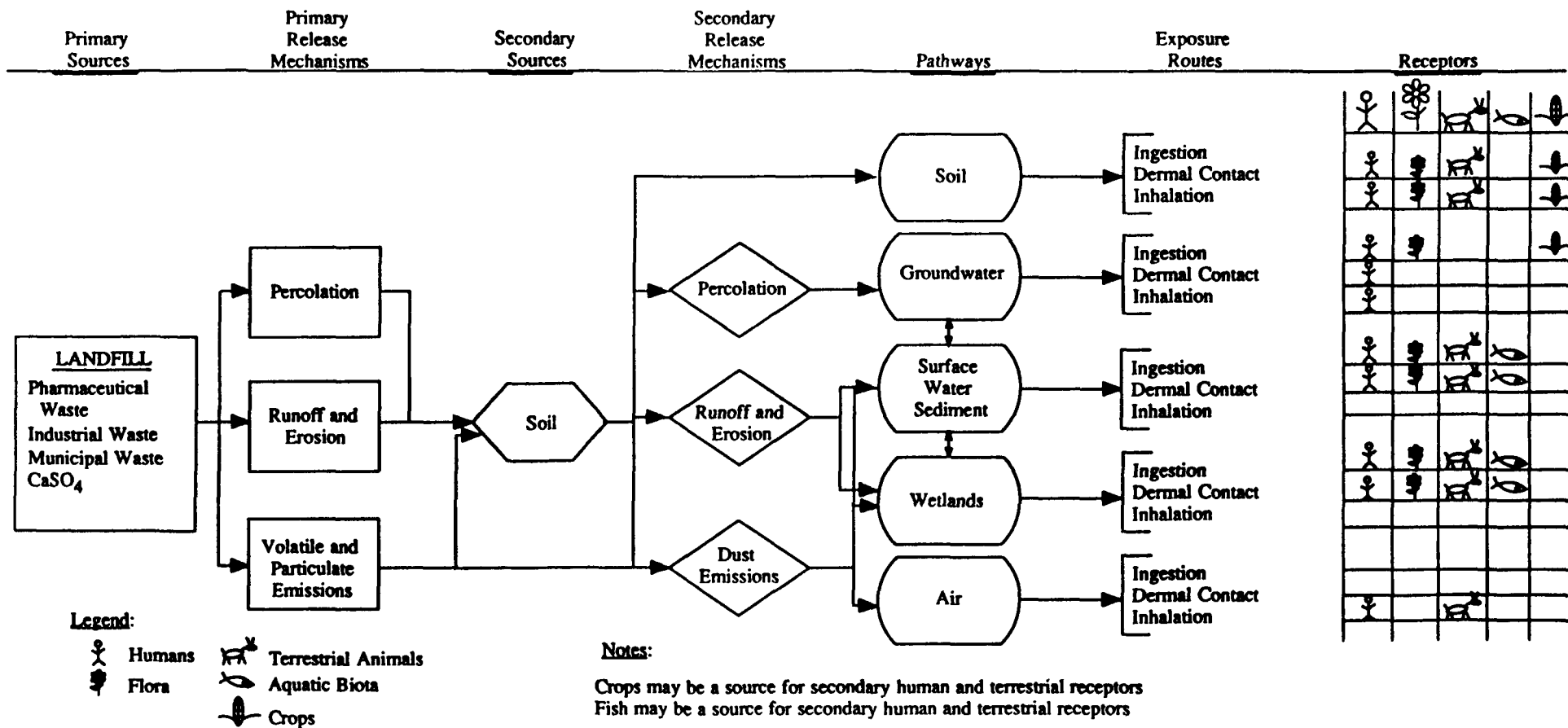


TABLE 3-1
DETECTED METALS - 1984
Himco Dump
Elkhart, Indiana

Units: ug/l (ppb)

Analytes	Field Blank	Upgradient Wells		E/17'	E/174'	M/24'	Dup M24'	P/24'	Offsite Down Gradient		Surface Water		Sediment	
		D/19'	D/174'						I/35'	I/172'	UG	DG	UG	DG
Aluminum	-	12,500	-	350,000	-	296	269	175	1,890	-	-	-	1,640	424
Arsenic	-	26	-	200	-	-	-	26	-	-	-	-	1.8	2.0
Barium	-	121	-	803	165	172	175	97	414	66	-	-	14	-
Beryllium	-	-	-	11	-	-	-	-	-	-	-	-	-	-
Cadmium	-	-	-	10	-	-	-	-	-	-	-	-	-	-
Chromium	-	370	-	461	-	16	12	-	-	-	-	-	3.9	1.3
Cobalt	-	-	-	132	-	-	-	-	-	-	-	-	2.7	-
Copper	-	73	-	555	-	-	-	-	-	-	-	-	3.9	-
Iron	-	67,400	1,230	146,000	1,580	12,300	14,800	11,400	5,520	507	246	210	4,380	1,550
Lead	-	73	-	401	-	7.7	9.0	6.7	-	-	-	-	5.8	1.6
Manganese	-	1,630	158	2,150	41	331	320	182	133	24	24	12	43	103
Mercury	-	0.21	-	1.4	-	-	-	-	-	-	-	-	-	-
Nickel	-	103	-	422	-	-	-	-	-	-	-	-	4.4	-
Selenium	-	-	2.0	14	-	-	-	4.7	-	-	-	-	-	-
Tin	-	-	-	-	32	-	-	-	-	55	-	-	1.1	1.2
Vanadium	-	-	-	326	-	-	-	-	-	-	-	-	-	-
Zinc	11	164	38	1,630	44	224	309	58	18	55	65	-	19	5

- = Not detected at or above contract required detection limit.

UG = Upgradient

DG = Downgradient

X/YY' = Well/sampling depth in feet.

DUP = Field Duplicate

ARCS/P/HIMCO/ABB

TABLE 3-1
DETECTED ORGANICS - 1984
Himco Dump
Elkhart, Indiana
(Continued)

Units: ug/l (ppb)

Compound	Field	Upgradient Wells		E/17'	E/174'	M/24'	Dup M/24'	P/24'	Offsite		Surface Water		Sediment	
	Blank	D/19'	D/174'						Down Gradient	I/35'	I/172'	UG	DG	Units: ug/kg (ppb)
<u>Volatiles</u>														
Acetone	32	39	39		164	60	100	230					492 C	66 C
Benzene	5 K	5 K	5		5 K	5 K	5 K	4	5 K		5 K	5 K		10 K
2-Butanone					106		79							
Chloroethane								13						
Chlorofluoromethane					43 J	57 J	37 J	18 J						
Dichlorodifluoromethane					61 J	79 J	56 J	14 J						
1,1-Dichloroethane								15						
Trans 1,2-Dichloroethene					8	9	7							
Diethylether								44 J					45 J	78 J
1,4-Dioxane								9 J						
Ethylbenzene									5 K					
2-Hexanone									5 K					
Methylene Chloride				5 K	5 K	5			5 K		15 C		319 C	249 C
Toluene	5 K			5 K	5 K	3	5 K	5 K	5 K	5 K			10 K	10 K
Trichloroethane					5 K		5 K	5 K						
Carbon Disulfide									1					
<u>Semi-Volatiles</u>														
1,2,4-Trichlorobenzene					10 K									
1,4-Dichlorobenzene					7 K									

K = Compound detected above instrument detection limit but below contract required detection limit.

J = Compound identified by computer library search, concentration estimated.

C = Associated lab blank contained detectable level, value reported has had blank level subtracted from it.

UG = Upgradient.

DG = Downgradient.

X/YY' = Well/sampling depth in feet.

DUP = Field Duplicate

TABLE 3-1
DETECTED ORGANICS - 1984
Himco Dump
Elkhart, Indiana
(Continued)

Units: ug/l (ppb)

<u>Semi-Volatiles</u>	<u>Field Blank</u>	<u>Upgradient Wells</u>		<u>E/17'</u>	<u>E/174'</u>	<u>M/24'</u>	<u>Dup M/24'</u>	<u>P/24'</u>	<u>Offsite Down Gradient</u>		<u>Surface Water</u>		<u>Sediment</u>	
		<u>D/19'</u>	<u>D/174'</u>						<u>1/35'</u>	<u>1/172'</u>	<u>UG</u>	<u>DG</u>	<u>UG</u>	<u>DG</u>
Naphthalene	10 K													
Di-N-Butylphthalate	10 K	10 K	10 K	10 K	15	10 K			10 K	10 K	10 K	10 K		
Acenaphthene					25 K									
2,4-Dinitrotoluene					20 K									
N-Nitrosodipropylamine					9									
Pyrene					13									
Phenol						62	76							
4-Methylphenol						197	235							
Bis(2-ethylhexyl) phthalate						10 K	266 C	20						
Diethylphthalate	10 K			10 K					10 K				150 K	
<u>Tentatively Identified Compounds</u>														
Caprolactam						224 J	145 J							
Sulfur						39 J	41 J						1180 J	
Dioctylester- hexanonedioic acid							1190 J							
<u>Unknowns</u>														
Volatile fraction							5 J							
Semi-Volatile fraction													1080 J	1390 J
<u>PCB/Pesticides</u>														
None detected														

K = Compound detected above instrument detection limit but below contract required detection limit.
J = Compound identified by computer library search, concentration estimated.
C = Associated lab blank contained detectable level, value reported has had blank level subtracted from it.
UG = Upgradient
DG = Downgradient
DUP = Field Duplicate

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surface water, sediment, and wetlands. Contamination of surface water, sediment, wetlands, and air could arise from the secondary release mechanism of dust emissions. Potential contaminant transport pathways to receptors include air, groundwater, sediment, surface water, and wetlands. Primary receptors include humans through ingestion, inhalation, and dermal contact; and terrestrial and aquatic environmental species through ingestion, inhalation, and dermal contact. Crops grown on the landfill cap could serve as a receptor through uptake (ingestion) of contaminated groundwater and surface water. Human and territorial receptors could then be exposed through ingestion and dermal contact.

The following contaminants of concern have been selected based on the 1984 historical data and on data needs for the risk assessment and the remedial alternative selection process:

Volatile Organics - CLP Target Compound List (TCL)
Semivolatile Organics - CLP Target Compound List (TCL)
PCBs/Pesticides - CLP Target Compound List (TCL)
Metals/Cyanide - CLP Target Analyte List (TAL)
Geotechnical Parameters - (grain size, Atterberg limits, permeability, consolidation, triaxial shear) Organic carbon will also be measured to assess the sorptive properties of site soils and existing cap materials for contaminant fate and transport determinations.

Water Quality Parameters

The specific water quality parameters to be measured and the rationale for their selection are listed below:

Chemical Oxygen Demand (COD): Measure of chemical oxidation in water; poorly degradable contaminants will elevate COD above BOD level.

Chloride: Major mobile anion associated with typical landfill leachate.

Sulfate: Anion associated with typical landfill leachate; reduction of organic sulfur occurs in anaerobic conditions and oxidation occurs in aerobic conditions.

Ammonia, Nitrate and Nitrite, Total Kjeldahl Nitrogen: Specification of nitrogen needed for remedial alternative selection.

Total Phosphorus: Indicator of agricultural use (on-site cropland); high levels enhance algae growth and may complicate some treatment alternatives.

Total Dissolved Solids, Total Suspended Solids: Remedial alternatives such as filtration and sorption are dependent on solids loading.

Alkalinity: Measure of buffering effect and ability to support algae growth; high levels may complicate some treatment alternatives.

Bromide: Mobile anion previously detected by USGS and selected as the indicator of the plume from the site.

Analysis of site samples for the water quality and geotechnical parameters will be accomplished through the CLP Special Analytical Services (SAS) program. Analysis of waste mass gas samples collected on sorbent tubes for TCL volatile organics will also be accomplished through the CLP SAS program. Detection limits for SAS analysis are included in the specific SAS requests in Appendix A. Quantitation limits for TAL metals/cyanide and TCL volatile organics, semivolatile organics, and PCBs/pesticides are contained in Tables 3-2 and 3-3. Quantitation limits for SAS analytes are contained in Table 3-4.

3.5 PROJECT OBJECTIVES

3.5.1 Objectives

The existing data base indicates that the primary potential contaminants associated with the Himco Dump Site are heavy metals and semivolatile and volatile organic compounds. The potentially contaminated media on-site include soil, surface water, sediment, and groundwater. Based on an initial evaluation and technical determinations reached at project scoping meetings, the primary objectives of the Himco Dump RI/FS are to:

- ° Characterize the nature and extent of contamination in the landfill, site soils, surface water, sediment, wetland remnant area, and groundwater. These media are potential contaminant sources or pathways.
- ° Determine the potential for contaminant transport from the landfill via air, groundwater, and sediment/surface water pathways.
- ° Evaluate a range of feasible, permanent solutions for on-site remediation in accordance with the Superfund Amendments and Reauthorization Act.
- ° Identify and develop standards and criteria for contaminant cleanup.
- ° Conduct a baseline human health evaluation and an environmental evaluation as appropriate for the site.

Phase I will determine if a source and a release exists and if a release via the groundwater pathway has occurred. If a source and release is indicated by the Phase I RI data, Phase II will further define the release and fill any data gaps for the ecological and human risk assessments, treatability studies, and remedial alternative evaluations.

TABLE 3-2

QUANTITATION LIMITS* FOR RAS TAL AND SAS INORGANIC COMPOUNDS
HIMCO DUMP RI/FS
Elkhart, Indiana

<u>Analyte</u>	Contract Required Detection Limit	Private Well	<u>Soil (mg/kg) (3)</u>
	<u>Water (ug/L) (1,2)</u>	<u>(ug/L)</u>	
Aluminum	200	100	40
Antimony	60	5	12
Arsenic	10	5	2
Barium	200	50	40
Beryllium	5	5	1
Cadmium	5	0.5	1
Calcium	5000	1000	1000
Chromium	10	10	2
Cobalt	50	10	10
Copper	25	10	5
Iron	100	100	20
Lead	3	2	1
Magnesium	5000	1000	1000
Manganese	15	10	3
Mercury	0.2	0.2	0.040
Nickel	40	20	8
Potassium	5000	2000	1000
Selenium	5	2	1
Silver	10	5	2
Sodium	5000	1000	1000
Thallium	10	2	2
Vanadium	50	10	10
Zinc	20	20	4
Cyanide	10	10	2

1. Subject to the restrictions specified in the first page of Part G, Section IV of Exhibit D (Alternate Methods - Catastrophic Failure) any analytical method specified in SOW Exhibit D may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Limit (CRDL) requirements. Higher detection limits may only be used in the following circumstance:

If the sample concentration exceeds five times the detection limit of the instrument or method in use, the value may be reported even though the instrument or method detection limit may not equal the Contract Required Detection Limit. This is illustrated in the example below:

TABLE 3-2

QUANTITATION LIMITS* FOR RAS TAL AND SAS INORGANIC COMPOUNDS
HIMCO DUMP RI/FS
Elkhart, Indiana
(Continued)

For lead:

Method in use - ICP

Instrument Detection Limit (IDL) - 40

Sample concentration - 220

Contract Required Detection Limit (CRDL) = 5

The value of 220 may be reported even though instrument detection limit is greater than CRDL. The instrument or method detection limit must be documented as described in Exhibit #.

2. The CRDL are the instrument detection limits obtained in pure water than must be met using the procedure in Exhibit E. The detection limits for samples may be considerably higher depending on the sample matrix.
3. The detection limits for soil samples are approximately 200 times that for water samples (per Region V Sample Handling Manual March, 1989).

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TABLE 3-3

QUANTITATION LIMITS* FOR RAS TCL AND SAS ORGANIC COMPOUNDS
HIMCO DUMP RI/FS
Elkhart, Indiana

<u>Volatiles</u>	<u>CAS Number</u>	<u>Water ug/L</u>	<u>Quantitation Limits**</u>	
			<u>Private Well ug/L</u>	<u>Low Soil/Sediment^a Ug/Kg</u>
1. Chloromethane	74-87-3	10	1.5	10
2. Bromomethane	74-83-9	10	1.5	10
3. Vinyl Chloride	75-01-4	2	1.5	10
4. Chloroethane	75-00-3	10	1.5	10
5. Methylene Chloride	75-09-2	5	1	5
6. Acetone	67-64-1	10	5	10
7. Carbon Disulfide	75-15-0	5	3	5
8. 1,1-Dichloroethene	75-35-4	5	1.5	5
9. 1,1-Dichloroethane	75-34-3	5	1.5	5
10. 1,2-Dichloroethene (total)	540-59-0	5	1.5	5
11. Chloroform	67-66-3	5	1.5	5
12. 1,2-Dichloroethane	107-06-2	5	1.5	5
13. 2-Butanone	78-93-3	10	5	10
14. 1,1,1-Trichloroethane	71-55-6	5	1.5	5
15. Carbon Tetrachloride	56-23-5	5	1.5	5
16. Vinyl Acetate	108-05-4	10	5	10
17. Bromodichloromethane	75-27-4	5	1.5	5
18. 1,2-Dichloropropane	78-87-5	5	1.5	5
19. cis-1,3-Dichloropropene	10061-01-5	5	2	5
20. Trichloroethene	79-01-6	5	1.5	5
21. Dibromochloromethane	124-48-1	5	1.5	5
22. 1,1,2-Trichloroethane	79-00-5	5	1.5	5
23. Benzene	71-43-2	5	1.5	5
24. trans-1,3-Dichloropropene	10061-02-6	5	1	5
25. Bromoform	75-25-2	5	1.5	5
26. 4-Methyl-2-pentanone	108-10-1	10	1.5	10
27. 2-Hexanone	591-78-6	10	5	10
28. Tetrachloroethene	127-18-4	5	1.5	5
29. Toluene	108-88-3	5	1.5	5
30. 1,1,2,2-Tetrachloroethane	79-34-5	5	1.5	5
31. Chlorobenzene	108-90-7	5	1.5	5
32. Ethyl Benzene	100-41-4	5	1.5	5
33. Styrene	100-42-5	5	1	5
34. Xylenes (Total)	1330-20-7	5	1.5	5

TABLE 3-3

QUANTITATION LIMITS* FOR RAS TCL AND SAS ORGANIC COMPOUNDS
HIMCO DUMP RI/FS
Elkhart, Indiana
(Continued)

Semi-Volatiles	CAS Number	Water ug/L	Quantitation Limits**	
			Private Well ug/L	Low Soil/Sediment ^b Ug/Kg
35. Phenol	108-95-2	10	2	330
36. bis(2-Chloroethyl) ether	111-44-4	10	1.5	330
37. 2-Chlorophenol	95-57-8	10	2	330
38. 1,3-Dichlorobenzene	541-73-1	10	2	330
39. 1,4-Dichlorobenzene	106-46-7	10	2	330
40. Benzyl alcohol	100-51-6	10	2	330
41. 1,2-Dichlorobenzene	95-50-1	10	2.5	330
42. 2-Methylphenol	95-48-7	10	1	330
43. bis(2-Chloroisopropyl) ether	108-60-1	10	2.5	330
44. 4-Methylphenol	106-44-5	10	1	330
45. N-Nitrosodipropylamine	621-64-7	10	1.5	330
46. Hexachloroethane	67-72-1	10	2	330
47. Nitrobenzene	98-95-3	10	2.5	330
48. Isophorone	78-59-1	10	2.5	330
49. 2-Nitrophenol	88-75-5	10	2	330
50. 2,4-Dimethylphenol	105-67-9	10	2	330
51. Benzoic acid	65-85-0	50	20	1600
52. bis(2-Chloroethoxy) methane	111-91-1	10	2.5	330
53. 2,4-Dichlorophenol	120-83-2	10	2	330
54. 1,2,4-Trichlorobenzene	120-82-1	10	2	330
55. Naphthalene	91-20-3	10	2	330
56. 4-Chloroaniline	106-47-8	10	2	330
57. Hexachlorobutadiene	87-68-3	10	2.5	330
58. 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	1.5	330
59. 2-Methylnaphthalene	91-57-6	10	2	330
60. Hexachlorocyclopentadiene	77-47-4	10	2	330
61. 2,4,6-Trichlorophenol	88-06-2	10	1.5	330
62. 2,4,5-Trichlorophenol	95-95-4	50	1.5	1600
63. 2-Chloronaphthalene	91-58-7	10	1.5	330
64. 2-Nitroaniline	88-74-4	50	1	1600
65. Dimethylphthalate	131-11-3	10	1.5	330
66. Acenaphthylene	208-96-8	10	1.5	330
67. 2,6-Dinitrotoluene	606-20-2	10	1	330
68. 3-Nitroaniline	99-09-2	50	2.5	1600
69. Acenaphthene	83-32-9	10	1.5	330

TABLE 3-3

QUANTITATION LIMITS* FOR RAS TCL AND SAS ORGANIC COMPOUNDS
HIMCO DUMP RI/FS
Elkhart, Indiana
(Continued)

<u>Semi-Volatiles</u>	<u>CAS Number</u>	<u>Water ug/L</u>	<u>Quantitation Limits**</u>	
			<u>Private Well ug/L</u>	<u>Low Soil/Sediment^b Ug/Kg</u>
70. 2,4-Dinitrophenol	51-28-5	50	(15)	1600
71. 4-Nitrophenol	100-02-7	50	1.5	1600
72. Dibenzofuran	132-64-9	10	1	330
73. 2,4-Dinitrotoluene	121-14-2	10	1	330
74. Diethylphthalate	84-66-2	10	1	330
75. 4-Chlorophenyl-phenyl ether	7005-72-3	10	1	330
76. Fluorene	86-73-7	10	1	330
77. 4-Nitroaniline	100-01-6	50	3	1600
78. 4,6-Dinitro-2-methylphenol	534-52-1	50	(15)	1600
79. N-nitrosodiphenylamine and Diphenylamine	86-30-6 and 122-39-4	10	1.5	330
80. 4-Bromophenyl-phenylether	101-55-3	10	1.5	330
81. Hexachlorobenzene	118-74-1	10	1.5	330
82. Pentachlorophenol	87-86-5	50	2	1600
83. Phenanthrene	85-01-8	10	1	330
84. Anthracene	120-12-7	10	2.5	330
85. Di-n-butylphthalate	84-74-2	10	2	330
86. Fluoranthene	206-44-0	10	1.5	330
87. Pyrene	129-00-0	10	1.5	330
88. Butylbenzylphthalate	85-68-7	10	3.5	330
89. 3,3'-Dichlorobenzidine	91-94-1	20	--	660
90. Benzo(a)anthracene	56-55-3	10	1.5	330
91. Chrysene	218-01-9	10	1.5***	330
92. bis(2-Ethylhexyl)phthalate	117-81-7	10	1	330
93. Di-n-octylphthalate	117-84-0	10	1.5	330
94. Benzo(b)fluoranthene	205-99-2	10	1.5	330
95. Benzo(k)fluoranthene	207-08-9	10	1.5****	330
96. Benzo(a)pyrene	50-32-8	10	2	330
97. Indeno(1,2,3-cd)pyrene	195-39-5	10	3.5	330
98. Dibenz(a,h)anthracene	53-70-3	10	2.5	330
99. Benzo(g,h,i)perylene	191-24-2	10	4	330

TABLE 3-3

QUANTITATION LIMITS* FOR RAS TCL AND SAS ORGANIC COMPOUNDS
HIMCO DUMP RI/FS
Elkhart, Indiana
(Continued)

<u>Pesticides/PCBs</u>	<u>CAS Number</u>	<u>Water</u> <u>ug/L</u>	<u>Quantitation Limits**</u>	
			<u>Private Well</u> <u>ug/L</u>	<u>Low Soil/Sediment^C</u> <u>Ug/Kg</u>
100. alpha-BHC	319-84-6	0.05	0.010	8.0
101. beta-BHC	319-85-7	0.05	0.005	8.0
102. delta-BHC	319-86-8	0.05	0.005	8.0
103. gamma-BHC (lindane)	58-89-9	0.05	0.005	8.0
104. Heptachlor	76-44-8	0.05	0.030	8.0
105. Aldrin	309-00-2	0.05	0.005	8.0
106. Heptachlor epoxide	1024-57-3	0.05	0.005	8.0
107. Endosulfan I	959-98-8	0.05	0.010	8.0
108. Dieldrin	60-57-1	0.10	0.010	16.0
109. 4,4'-DDE	72-55-9	0.10	0.005	16.0
110. Endrin	72-20-8	0.10	0.010	16.0
111. Endosulfan II	33213-65-9	0.10	0.010	16.0
112. 4,4'-DDD	72-54-8	0.10	0.020	16.0
113. Endosulfan sulfate	1031-07-8	0.10	0.10	16.0
114. 4,4'-DDT	50-29-3	0.10	0.020	16.0
115. Methoxychlor	72-43-5	0.5	0.020	80.0
116. Endrin ketone	53494-70-5	0.10	0.030	16.0
117. alpha-Chlordane	5103-71-9	0.5	0.020	80.0
118. gamma-Chlordane	5103-74-2	0.5	0.020	80.0
119. Toxaphene	8001-35-2	1.0	0.25	160.0
120. Aroclor-1016	12674-11-2	0.5	0.10	80.0
121. Aroclor-1221	11104-28-2	0.5	0.10	80.0
122. Aroclor-1232	11141-16-5	0.5	0.10	80.0
123. Aroclor-1242	53469-21-9	0.5	0.10	80.0
124. Aroclor-1248	12672-29-6	0.5	0.10	80.0
125. Aroclor-1254	11097-69-1	1.0	0.10	160.0
126. Aroclor-1260	11096-82-5	1.0	0.10	160.0

^a Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for Volatile TCL Compounds are 125 times the individual Low Soil/Sediment CRQL.

^b Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for Semi-Volatile TCL Compounds are 60 times the individual Low Soil/Sediment CRQL.

^c Medium Soil/Sediment Contract Required Quantitation Limits (CRQL) for Pesticide/PCB TCL compounds are 15 times the individual Low Soil/Sediment CRQL.

- * Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.
- ** Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.
- *** Reported with benzo(a)anthracene.
- **** Reported with benzo(b)fluoranthene.
- () Estimated detection limit, actual values under investigation.

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TABLE 3-4

QUANTITATION LIMITS FOR SAS ANALYTES
HIMCO DUMP RI/FS
Elkhart, Indiana

<u>SAS Analyte</u>	<u>Matrix</u>	<u>Quantitation Limit</u>
Chloride	groundwater	5 mg/L
Sulfate	groundwater	5 mg/L
TDS	groundwater	20 mg/L
TSS	groundwater	2-3 mg/L
Alkalinity	groundwater	2 mg/L
TP	groundwater	0.05 mg/L
TKN	groundwater	0.1 mg/L
NH ₃	groundwater	0.1 mg/L
NO ₂ & NO ₃	groundwater	0.10 mg/L
COD	groundwater	5 mg/L
Bromide	groundwater	0.10 mg/L
Organics -		
Residential Wells - See Table 3-3		
Inorganics -		
Residential Wells - See Table 3-2		
Atterberg Limits	soil	not applicable
Particle Size	soil	1%
Permeability	soil	not applicable
Consolidation	soil	not applicable
Triaxial Shear	soil	not applicable
Organic Carbon	soil	0.10%

TABLE 3-4

QUANTITATION LIMITS FOR SAS ANALYTES
HIMCO DUMP RI/FS
Elkhart, Indiana
(Continued)

<u>Volatile Organics</u>	<u>Matrix</u>	<u>Quantitation Limit (ng/tube)**</u>
Benzene	sorbent tube	5
Bromodichloromethane	sorbent tube	5
Bromoform	sorbent tube	5
Bromomethane	sorbent tube	5
Carbon Tetrachloride	sorbent tube	5
Chlorobenzene	sorbent tube	5
Chloroethane	sorbent tube	5
Chloroform	sorbent tube	5
Chloromethane	sorbent tube	5
Dibromochloromethane	sorbent tube	5
1,1-Dichloroethane	sorbent tube	5
1,2-Dichloroethane	sorbent tube	5
1,1-Dichloroethene	sorbent tube	5
1,2-Dichloroethene (Total)	sorbent tube	5
1,2-Dichloropropene	sorbent tube	5
Cis-1,3-Dichloropropene	sorbent tube	5
Trans-1,3-Dichloropropene	sorbent tube	5
Ethyl Benzene	sorbent tube	5
Methylene Chloride	sorbent tube	5
1,1,2,2-Tetrachloroethane	sorbent tube	5
Tetrachloroethene	sorbent tube	5
Toluene	sorbent tube	5
1,1,1-Trichloroethane	sorbent tube	5
1,1,2-Trichloroethane	sorbent tube	5
Trichloroethene	sorbent tube	5
Vinyl Chloride	sorbent tube	5
Acetone	sorbent tube	5
Carbon Disulfide	sorbent tube	5
2-Butanone	sorbent tube	5
Vinyl Acetate	sorbent tube	5
4-Methyl-2-Pentanone	sorbent tube	5
2-Hexanone	sorbent tube	5
Styrene	sorbent tube	5
m-Xylene	sorbent tube	5
o-Xylene	sorbent tube	5
p-Xylene	sorbent tube	5

** Assuming cartridge is packed with 2 grams of Tenax.

The Phase I field investigation will consist of the following tasks:

- Site survey and topographic mapping.
- Electromagnetic survey for fill boundary determination.
- Sampling and analysis of wetlands.
- Monitoring well installation, sampling and analysis (quarterly for four quarters).
- Soil boring sampling and analysis.
- Existing monitoring well sampling and analysis (quarterly for four quarters)
- Private well sampling and analysis (quarterly for four quarters).
- Leachate sampling and analysis (if seeps present).
- Landfill waste sampling and geotechnical analysis.
- Landfill cap surface soil sampling and analysis.
- Landfill waste mass gas sampling and analysis.
- Sediment and surface water sampling and analysis from quarry.
- Sediment and surface water sampling and analysis from fish ponds.
- Installation of staff gauges.

The scope of the Phase II field investigation will be dependent on whether a source exists and releases to groundwater, surface water, sediment, and air have occurred. The following tasks are proposed for Phase II if a source and releases are determined by the Phase I RI data:

- Gravity survey to map bedrock valley location.
- Sampling and analysis of quarry biota.
- Sampling and analysis of pond biota.
- Air pathway analysis.

The rationale for each of the tasks and a summary of the field procedures and chemical analyses are discussed below. Detailed descriptions of exact sample locations and specific sample collection protocols are included in the Field Sampling Plan, Volume 2.

3.5.2 Data Uses

The data collected during the RI will be used to satisfy data needs associated with site characterization, risk assessment, and evaluation of remedial alternatives during the FS. Data needs specific to the Himco Dump RI/FS have been identified by evaluating existing data with reference to the Conceptual Site Model and are discussed in Section 4.0 of the Work Plan (Volume 1A).

Due to continuing concerns from the nearby residents who have not been using their residential well water for drinking or cooking purposes, residential wells will be sampled and analyzed quarterly using low level detection limits to determine if the well water presents a risk to health and meets current state and federal criteria. Data from the residential wells will also be used to determine if a plume from the landfill exists.

Users of the data generated from this RI/FS will consist of the EPA, IDEM, health assessment scientists, engineers, hydrogeologists, geologists, biologists, and chemists. The potentially responsible parties (PRPs) will also review the data collected. Wetlands were determined to be present by the U.S. Fish and Wildlife Service, Bloomington, Indiana. A 404 permit may be required, and the endangerment assessment will be reviewed by the Corps of Engineers since the EPA will not be funding the remedial design. The U.S. Fish and Wildlife Service will also be involved in the endangerment assessment. Because the Himco Dump is proposed for the National Priorities List (NPL), the RI data will also be used by the EPA to determine whether to list or delist the site.

3.5.3 Data Quality Objectives

Data Quality Objectives (DQOs) were developed by EPA based on the premise that different end-uses of data necessitate varying levels of analytical data quality. EPA has defined five levels of analytical data quality: Level (I), field screening; Level (II), on-site analysis; Level (III), off-site analysis by EPA-approved method; Level (IV), Contract Laboratory Program (CLP) Routine Analytical Services (RAS) analysis; and Level (V), Special Analytical Services (SAS) analysis.

Data needs specific to the Himco Dump RI/FS have been identified by evaluating existing data with reference to the Conceptual Site Model and determining what additional data are necessary in order to accomplish the project objectives. The RI field program will key in on the sampling and analysis of the suspected primary source and the pathways as presented in the Conceptual Site Model.

The primary suspected contaminant source at the Himco Dump Site is the landfill. No direct analysis of the composition and concentrations of the fill or leachate is available. The type, depth, and amount of waste disposed of at the site is unknown. Therefore, no straight-forward statistical method is available for determining the expected variations or sampling density required to obtain a representative estimate of the contamination. Instead, a sampling grid will be established on the fill on 250-foot centers to provide a systematic coverage of the fill area. The limits of fill will be determined by an electromagnetic survey. Soil/waste samples will then be collected for chemical analysis (DQO Level IV) at the surface (0 to 6 inches) to determine if risk to receptors by particulate emission releases exists and to evaluate remedial alternatives. Geotechnical analyses will be done on samples collected to a depth of 3 feet in the waste. DQO Level V will be used for the geotechnical parameters of consolidation and triaxial shear. These geotechnical parameters are needed to predict the amount and rate of settlement of the fill under loads and to determine the strength of the existing cap for remedial alternative selection.

Field instruments will be used to measure the percent methane and hydrogen sulfide at the grid sampling points using DQO Level I. A field portable photoionization detector will be used to provide data on the order of magnitude of VOCs at each of the borings (DQO Level I). Selected grid locations with the highest field VOC concentrations will be sampled using a calibrated pump and sorbent tubes. Samples will be analyzed using DQO Level V to determine the identity and quantity of VOCs in the waste mass gas for purposes of risk assessment and remedial alternative selection. If leachate seeps are visible during the Phase I field program, they will be sampled and analyzed using DQO Levels IV and V.

The secondary contaminant source area at the Himco Dump Site is the soil. Based on the measured contamination in groundwater immediately downgradient of the site, the vadose zone soil is believed to be contaminated. However, no direct analysis of any site soils has been done. Deeper subsurface drilling through the waste to sample underlying soil is not proposed for Phase I RI activities. However, during installation of the ten proposed new monitoring wells, soil samples will be collected and analyzed using DQO Level IV. Selected soil samples will also be analyzed for the geotechnical parameters of Atterberg limits, grain size, total organic carbon and permeability using DQO Level V to characterize the geologic conditions.

Four potential contaminant migration pathways have been identified in the Conceptual Site Model. Groundwater is the pathway of greatest concern as site-derived contaminants have been measured historically and receptors are located immediately downgradient of the site. Groundwater from both existing USGS wells and proposed new wells will be collected and analyzed quarterly using DQO Level IV.

The groundwater chemistry data will be used to determine the nature and extent of contamination and develop the risk assessment. In order to fully evaluate remedial alternatives, hydrogeologic data also need to be collected. Aquifer conductivity will be determined using slug tests on new and existing wells. Water levels will be measured to develop a groundwater table map. Groundwater discharge to surface water bodies (quarry, fish ponds) will also be assessed with staff gauges. Field parameters that assess groundwater stability after purging (pH, conductivity, dissolved oxygen (DO), and temperature) will be measured using DQO Level I.

Private well sampling will be conducted quarterly at the six residences that are located along the southern edge of the site. Historical state data indicated elevated levels of the water quality parameters of COD, iron, and manganese in the shallow wells on these properties. No validated chemistry data are available on the deeper wells. DQO Level V will be used in the analysis of the private well samples, since lower than the CLP RAS detection limits are needed to meet Maximum Contaminant Levels (MCLs) and Recommended Maximum Contaminant Levels (RMCLs) for drinking water. Field parameters of pH, conductivity, dissolved oxygen (DO), and temperature will be measured using DQO Level I to assess groundwater stability after purging.

The surface water and sediment pathway will be sampled during the Phase I RI using DQO Levels IV and V. Two fish ponds and a quarry are located adjacent to the fill area and may be impacted from the site. No historical chemical data exist for the surface water or sediment in the quarry or ponds. Data will be used in developing the ecological and human risk assessment.

A determination of the presence of wetlands as defined by the Federal Manual for Identifying and Delineating Jurisdictional Wetlands was made by the U.S. Fish and Wildlife Service, Bloomington, Indiana. Sampling of wetland soils will therefore occur in Phase I.

The air pathway will be modeled using data on VOCs collected from the DQO Level V waste mass gas monitoring during Phase I. No direct air monitoring and sampling is planned for Phase II unless data from Phase I indicate a significant risk to receptors.

A summary of the DQO levels proposed for this investigation is presented in Table 3-5. DQO summary forms used during the scoping process are included in Appendix B.

3.6 SAMPLE NETWORK DESIGN AND RATIONALE

A sampling and analysis summary table is presented in Table 3-6. Complete descriptions of sampling activities including sampling location diagrams, number of samples, and rationale of selected locations are presented in the Field Sampling Plan (FSP) (Volume 2).

3.7 PROJECT SCHEDULE

The proposed schedule is presented in Chapter 8 of the Work Plan (Volume 1A).

The proposed schedule for the RI/FS is based on conditions discussed in the Work Plan. A six-week CLP turnaround time for RAS and SAS sample analysis with an additional three-week turnaround time for EPA data validation were assumed in developing this schedule. Groundwater and private well water samples will be collected quarterly (every 3 months) and sent through the CLP for one year to collect enough data to assess if seasonal variations are occurring.

The proposed schedule assumes ready access to the site. The proposed schedule also assumes that health and safety personnel protection requirements are Level D, with possible upgrade to Level C, as detailed in the Himco Dump RI/FS Health and Safety Plan (Volume 4). Variation from these assumptions may result in schedule delays.

TABLE 3-5

DQO LEVELS
HIMCO DUMP RI/FS
Elkhart, Indiana

<u>Field Analysis</u>	<u>Matrix</u>				
	<u>Landfill</u> <u>Cap soil/Waste</u>	<u>Waste Mass</u> <u>Gas</u>	<u>Groundwater</u> <u>Surface</u> <u>Water</u>	<u>Soil</u> <u>Sediment</u>	<u>Residential</u> <u>Wells</u>
pH			I		I
Conductivity			I		I
Dissolved oxygen			I		I
Temperature			I		I
% Methane		I			
% Hydrogen sulfide		I			
Gas Volume		I			
Volatile organics by HNu	I	I		I	
Electromagnetic survey	I				
Slug test/staff gauge			I		
Water Level			I		
Radioactivity	I			I	
<u>Laboratory Analysis</u>					
Routine Analytical Services:					
TCL volatiles	IV		IV	IV	
TCL semi-volatiles	IV		IV	IV	
TCL PCB/pesticides	IV		IV	IV	
TAL metals/CN	IV		IV	IV	
Special Analytical Services:					
TCL volatiles - sor bent tubes		V			
Water quality			V		V
Organic Carbon				V	
Grain size	V			V	
Atterberg limits	V			V	
Permeability				V	
Consolidation	V				
Triaxial shear	V				
TCL volatiles					V
TCL semi-volatiles					V
TCL PCB/pesticides					V
TAL metals/CN					V

TABLE -6

**Sampling and Analysis Summary Table for Himco Dump
Phase I**

Sample Matrix	Field Parameters	DQO Level	Lab Parameters	DQO Level	Lab	Field Samples	Field QC						Lab QC			Purpose of Samples
							BB ³	BG	TB**	FB	FD	Total to Lab	LD	MSD*	MS	
SITE CONDITIONS	EM-31 Conductivity	I					-	-	-	-	-					Determine limits of fill Assess if buried drums present
LANDFILL CAP AND WASTE	Radioactivity VOAs by IINU	I 6-18"	<u>Geotech.</u> Consolidation ¹ Triaxial Shear ¹ Grain Size ² Atterberg Limits ²	V V V V	CLP SAS	2 Max 5 Max 5 Max 5 Max	-	-	-	-	-	2 Max 5 Max 5 Max 5 Max	-	-	-	Use to predict amount and rate of settlement under loads for remedial alternatives Determine strength of waste for remedial alternatives Determine material properties of waste mass
			TCL VOA	IV	CLP	12	-	-	-	-	1	13	-	1	1	Risk assessment - dust particulate emission releases Evaluate remedial alternatives
			TCL IINA	IV	CLP	12	-	-	-	-	1	13	-	1	1	
			TCL PCB/P	IV	CLP	12	-	-	-	-	1	13	-	1	1	
			TAL Metals/CN	IV	CLP	12	-	-	-	-	1	13	1	-	1	
LANDFILL WASTE MASS GAS	Methane	I	TCL VOA (Sorbent tubes)	V	CLP SAS	10 Max	1 ⁴	1 ⁵	1	-	1	13 Max	-	1	1	Waste characterization. Risk assessment of volatile emission release mechanism Evaluate remedial alternatives Evaluate sampling rate of pump
	Hydrogen Sulfide	I														
	VOCs by IINU	I	TCL VOA (Sorbent Tubes)	V	Quick Turn SAS	2	-	-	1	-	-	2	-	1	1	

NOTES:

¹Thin-walled Shelby tube

²Ziploc bag

³Assume bottle cleaning protocol submitted with QAPP acceptable

⁴Unexposed sorbent tube

⁵Offsite location

* MS/MSD samples required for organic analysis. Aqueous samples (groundwater, surface water, leachate, private well water) shall be collected, with extra sample volume at a frequency of one per 20 or fewer investigative samples. Triple the normal volume will be collected for VOAs and double the normal volume will be collected for BNA and PCB/P.

** Trip blanks will be shipped at a frequency of one per cooler of aqueous/sorbent tubes containing aqueous/sorbent tube samples for VOA analysis.

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							BB ¹	BG	TB**	FB	FD	Total to Lab	LD	MSD*	MS	
LEACHATE (if available)	pH	1	TCL VOA	IV	CLP	4 Max	-	-	1	1	1	6	-	1	1	Waste characterization of primary source Risk assessment Evaluate remedial alternatives
	Conductivity	1	TCL BNA	IV	CLP	4 Max	-	-	-	1	1	6	-	1	1	
	DO	1	TCL PCB/P	IV	CLP	4 Max	-	-	-	1	1	6	-	1	1	
	Temperature	1	TAL Metals/CN (Total) ³	IV	CLP	4 Max	-	-	-	1	1	6	1	-	1	
			Water Quality ⁴	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			COD	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			Cl	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			SO ₄	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			NH ₃	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			NO ₂ +NO ₃	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			TKN	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			TP	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			TDS	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			TSS	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			alkalinity	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	
			bromide, dissolved	V	CLP SAS	4 Max	-	-	-	1	1	6	1	-	1	

NOTES:

¹Assume bottle cleaning protocol submitted with QAPP acceptable.

²Assume able to access 6 abandoned shallow wells.

³Total metals are defined as digestion and analysis of TAL metals on an unfiltered sample. Dissolved metals are defined as digestion and analysis of TAL metals on a sample filtered in the field.

⁴Water quality analyses will be done on unfiltered sample except for bromide which will be field filtered.

Page 2 of 7

* MS/MSD samples required for organic analysis. Aqueous samples (groundwater, surface water, leachate, private well water) shall be collected, with extra sample volume at a frequency of one per 20 or fewer investigative samples. Triple the normal volume will be collected for VOAs and double the normal volume will be collected for BNA and PCB/P.

** Trip blanks will be shipped at a frequency of one per cooler of aqueous/sorbent tubes containing aqueous/sorbent tube samples for VOA analysis.

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MSD = Matrix Spike Duplicate

MS = Matrix Spike

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UG = Upgradient

TABLE 3-6
Sampling and Analysis Summary Table for Himco Dump
Phase I

Sample Matrix	Field Parameters	DQO Level	Lab Parameters	DQO Level	Lab	# Field Samples	Field QC						Lab QC			Purpose of Samples
							BB ¹	BG	TB**	FB	FD	Total to Lab	LD	MSD*	MS	
EXISTING USGS WELL WATER	Slug Test	I	TCL VOA	IV	CLP	18	-	5 UG	2	2	2	27	-	2	2	Determine nature and extent of contamination Develop groundwater table map Evaluate remedial alternatives Risk assessment
	Water Level	I	TCL BNA	IV	CLP	18	-	5 UG	-	2	2	27	-	2	2	
	pH	I	TCL PCB/P	IV	CLP	18	-	5 UG	-	2	2	27	-	2	2	
	Conductivity	I	TAL Metals/CN (Total and Dissolved) ³	IV	CLP	18	-	5 UG	-	2	2	27	2	-	2	
	DO	I	Water Quality ⁴	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	
	Temperature	I	CO ₂	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	
			Cl	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	
			SO ₄	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	
			NO ₃	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	
			NO ₂ /NO ₃	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	
			TKN	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	
			TP	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	
			TDS	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	-	
			TSS	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	-	
			alkalinity	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	
			bromide, dissolved	V	CLP SAS	18	-	5 UG	-	2	2	27	3	-	3	

NOTES:

¹Assume bottle cleaning protocol submitted with QAPP acceptable.

²Assume able to access 6 abandoned shallow wells.

³Total metals are defined as digestion and analysis of TAL metals on an unfiltered sample. Dissolved metals are defined as digestion and analysis of TAL metals on a sample filtered in the field.

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Page 3 of 7

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** Trip blanks will be shipped at a frequency of one per cooler of aqueous/sorbent tubes containing aqueous/sorbent tube samples for VOA analysis.

† Groundwater chemical samples will be taken quarterly. Four sets of the same number of samples given above will be taken.

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12/11/04 HIMCO

TABLE 3-6
Sampling and Analysis Summary Table for Himco Dump
Phase I

Sample Matrix	Field Parameters	DQO Level	Lab Parameters	DQO Level	Lab	#Field Samples	Field QC						Lab QC			Purpose of Samples
							BB ¹	BG	TB**	FB	FD	Total to Lab	LD	MSD*	MS	
PRIVATE WELL WATER	pH Conductivity DO Temperature	1	TCL VOA	V	CLP SAS	12 Max ³	-	-	1	2	2	16	-	1	1	Determine nature and extent of contamination Evaluate remedial alternatives Risk Assessment
			TCL BNA	V	CLP SAS	12 Max ²	-	-	-	2	2	16	-	1	1	
			TCL PCB/P	V	CLP SAS	12 Max ²	-	-	-	2	2	16	-	1	1	
			TAL Metals/CN (Total) ³	V	CLP SAS	12 Max ²	-	-	-	2	2	16	1	-	1	
			Water Quality ⁴	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	
			COD	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	
			Cl	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	
			SO ₄	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	
			NH ₃	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	
			NO ₂ /NO ₃	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	
			TKN	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	
			TP	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	
			TDS	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	-	
			TSS	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	-	
			alkalinity	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	
			bromide, dissolved	V	CLP SAS	12 Max ²	-	-	-	2	2	16	2	-	2	

NOTES:

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²Assume able to access 6 abandoned shallow wells.

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Page 4 of 7

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2-11-18 JRM/CTM

TABLE 3-6
Sampling and Analysis Summary Table for Himco Dump
Phase I

Sample Matrix	Field Parameters	DQO Level	Lab Parameters	DQO Level	Lab	#Field Samples	Field QC						Lab QC			Purpose of Samples
							BB ¹	BG	TB**	FB	FD	Total to Lab	LD	MSD ³	MS	
SOIL FROM NEW WELL INSTALLATIONS	VOAs by HNU Radioactivity	1	TCL VOA	IV	CLP	30 Max	-	-	-	-	3	33	-	2	2	Determine subsurface geology and soil chemistry for evaluation of remedial alternatives Nature and extent of contamination
		1	TCL BNA	IV	CLP	30 Max	-	-	-	-	3	33	-	2	2	
			TCL PCB/P	IV	CLP	30 Max	-	-	-	-	3	33	-	2	2	
			TAL Metals/CN	IV	CLP	30 Max	-	-	-	-	3	33	2	-	2	
			Geotech TOC	V	CLP SAS	20 Max	-	-	-	-	-	20				
			Atterberg	V	CLP SAS	20 Max	-	-	-	-	-	20				
			Grain Size Permeability	V	CLP SAS	20 Max	-	-	-	-	-	20				
NEW WELLS GROUNDWATER	Slug Test Water Level pH Conductivity DO Temperature	1	TCL VOA	IV	CLP	7	-	3UG	1	1	1	12	-	1	1	Determine if USGS wells suitable for chemistry Nature and extent of contamination Risk assessment
		1	TCL BNA	IV	CLP	7	-	3UG	-	1	1	12	-	1	1	
		1	TCL PCB/P	IV	CLP	7	-	3UG	-	1	1	12	-	1	1	
		1	TAL Metals/CN (Total and Dissolved) ²	IV	CLP	7	-	3UG	-	1	1	12	1	-	1	
			Water Quality ³	V	CLP SAS	7	-	3UG	-	1	1	12	2	-	2	
			COD	V	CLP SAS	7	-	3UG	-	1	1	12	2	-	2	
			Cl	V	CLP SAS	7	-	3UG	-	1	1	12	2	-	2	
			SO ₄	V	CLP SAS	7	-	3UG	-	1	1	12	2	-	2	
			NH ₄	V	CLP SAS	7	-	3UG	-	1	1	12	2	-	2	
			NO ₂ /NO ₃	V	CLP SAS	7	-	3UG	-	1	1	12	2	-	2	

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REVISIONS

TABLE 3-6
Sampling and Analysis Summary Table for Himco Dump
Phase I

Sample Matrix	Field Parameters	DQO Level	Lab Parameters	DQO Level	Lab	Field Samples	Field QC						Lab QC			Purpose of Samples
							BB ¹	BG	TB**	FB	FD	Total to Lab	LD	MSD*	MS	
NEW WELLS GROUNDWATER (CONTINUED)			TKN	V	CLP SAS	7	-	3 UG	-	1	1	12	2	-	2	
			TP	V	CLP SAS	7	-	3 UG	-	1	1	12	2	-	2	
			TDS	V	CLP SAS	7	-	3 UG	-	1	1	12	2	-	-	
			TSS	V	CLP SAS	7	-	3 UG	-	1	1	12	2	-	-	
			alkalinity	V	CLP SAS	7	-	3 UG	-	1	1	12	2	-	2	
			bromide, dissolved	V	CLP SAS	7	-	3 UG	-	1	1	12	2	-	2	
SURFACE WATER (QUARRY, FISH PONDS)	Staff Gauge	1	TCL VOA	IV	CLP	12	-	-	1	2	2	16	-	1	1	Ecological and human risk assessment
	pH	1	TCL BNA	IV	CLP	12	-	-	-	2	2	16	-	1	1	
	Conductivity	1	TCL PCB/P	IV	CLP	12	-	-	-	2	2	16	-	1	1	
	DO	1	TAL Metals/CN (Total)	IV	CLP	12	-	-	-	2	2	16	1	-	-	
	Temperature	1	Water Quality ³	V	CLP SAS	12	-	-	-	2	2	16	1	-	1	
			COD	V	CLP SAS	12	-	-	-	2	2	16	2	-	2	
			Cl	V	CLP SAS	12	-	-	-	2	2	16	2	-	2	
			SO ₄	V	CLP SAS	12	-	-	-	2	2	16	2	-	2	
			NH ₄	V	CLP SAS	12	-	-	-	2	2	16	2	-	2	
			NO ₂ /NO ₃	V	CLP SAS	12	-	-	-	2	2	16	2	-	2	
			TKN	V	CLP SAS	12	-	-	-	2	2	16	2	-	2	
			TP	V	CLP SAS	12	-	-	-	2	2	16	2	-	2	
			TDS	V	CLP SAS	12	-	-	-	2	2	16	2	-	-	
			TSS	V	CLP SAS	12	-	-	-	2	2	16	2	-	-	
			alkalinity	V	CLP SAS	12	-	-	-	2	2	16	2	-	2	
			bromide, dissolved	V	CLP SAS	12	-	-	-	2	2	16	2	-	2	

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8 KUTERAHIMCO6

TABLE 3-6
Sampling and Analysis Summary Table for Himco Dump
Phase I

Sample Matrix	Field Parameters	DQO Level	Lab Parameters	DQO Level	Lab	Field Samples	Field QC						Lab QC			Purpose of Samples
							BB ¹	BG	TB**	FB	FD	Total to Lab	LD	MSD*	MS	
SEDIMENT (QUARRY, FISH POND, WETLANDS)			TCL VOA	IV	CLP	12	-	-	-	-	2	14	-	1	1	Ecological and human risk assessment
			TCL BNA	IV	CLP	12	-	-	-	-	2	14	-	1	1	
			TCL PCB/P	IV	CLP	12	-	-	-	-	2	14	-	1	1	
			TAL Metals/CN	IV	CLP	12	-	-	-	-	2	14	1	-	1	
WETLAND SOIL (GRAVEL PIT AREA (6), WETLAND REMNANT (4), OFF-SITE NW AREA (6))			TCL VOA	IV	CLP	16	-	-	-	-	2	18	-	1	1	Ecological and human risk assessment
			TCL BNA	IV	CLP	16	-	-	-	-	2	18	-	1	1	
			TCL PCB/P	IV	CLP	16	-	-	-	-	2	18	-	1	1	
			TAL Metals/CN	IV	CLP	16	-	-	-	-	2	18	1	-	1	
RESIDENTIAL BASEMENT AIR	Methane Hydrogen Sulfide	1 1														Determine if landfill gas migrating off-site

NOTES:

¹ Assume bottle cleaning protocol submitted with QAPP acceptable.

* MS/MSD samples required for organic analysis. Aqueous samples (groundwater, surface water, leachate, private well water) shall be collected, with extra sample volume at a frequency of one per 20 or fewer investigative samples. Triple the normal volume will be collected for VOAs and double the normal volume will be collected for BNA and PCB/P.

** Trip blanks will be shipped at a frequency of one per cooler of aqueous/sorbent tubes containing aqueous/sorbent tube samples for VOA analysis.

Legend

BB = Bottle Blank
 BG = Background Blank
 TB = Trip Blank
 FB = Field Blank
 FD = Field Duplicate
 LD = Lab Duplicate
 MSD = Matrix Spike Duplicate
 MS = Matrix Spike
 - = Not Applicable
 UG = Upgradient

4.0 PROJECT ORGANIZATION AND RESPONSIBILITY

At the direction of the Region V Remedial Project Manager (RPM), with final authority by the Region V Regional Project Officer (RPO), Donohue has overall responsibility for the RI/FS. Donohue will perform the field investigations and prepare the RI and FS reports. A project organization chart is presented in Figure 4-1. Specific duties of field team members are described in Section 3.0 of the FSP.

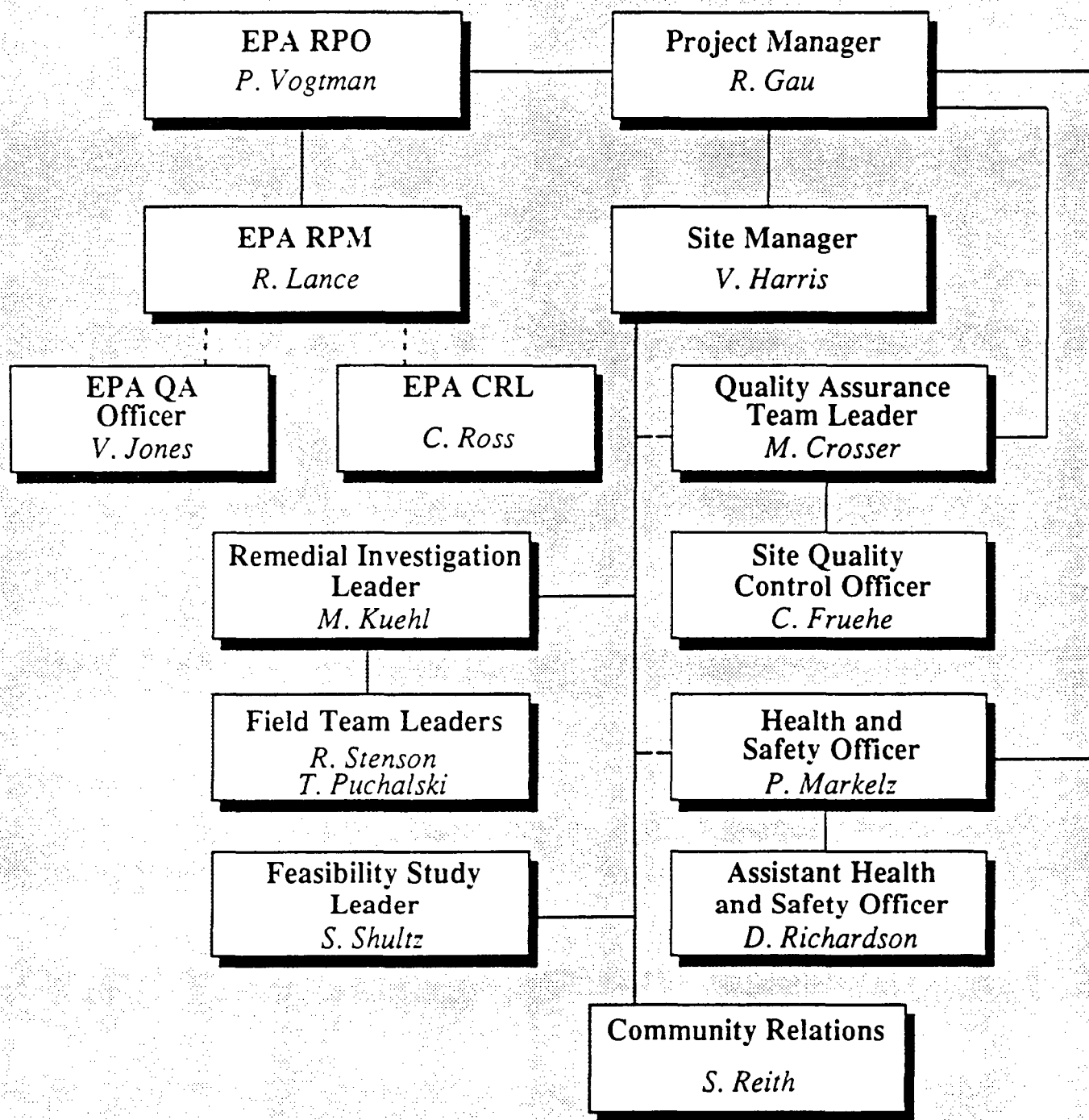
4.1 MANAGEMENT RESPONSIBILITIES

Operational responsibilities involving execution and direct management of the technical and administrative aspects of this project have been assigned as follows:

- ° Patricia Vogtman
Regional Project Officer (RPO)
U.S. EPA Region V
- ° Robert Lance
Remedial Project Manager (RPM)
U.S. EPA Region V
- ° Roman Gau
ARCS Project Manager (PM)
Donohue & Associates, Inc.
- ° Vanessa A. Harris
Site Manager (SM)
Donohue & Associates, Inc.

4.2 QA ORGANIZATION

<u>Tasks</u>	<u>Responsible Organization/Personnel</u>
° Final review and approval of RI/FS QAPP	U.S. EPA Region V RPM U.S. EPA Region V QA Officer
° QA program for CLP; RAS/SAS performance and systems audits for CLP RAS/SAS including evidence audits	U.S. EPA Hdqtrs. OERR U.S. EPA Sample Mgmt. Office U.S. EPA Region V CRL LSSS U.S. EPA EMSL - Las Vegas, QA Div. NEIC Evidence Audit Team (Techlaw, Inc.)



Donohue

June 1990

PROJECT ORGANIZATION

HIMCO DUMP
ELKHART, INDIANA

Engineers ■ Architects ■ Scientists

FIGURE 4-1

- ° Internal QA review and approval of reports, SOPs, and field activities; identifying and documenting non-conformance and corrective actions. Donohue Technical Services/Quality Assurance Manager (TSQAM)
- ° Evidence audits of field records Donohue TSQAM and NEIC Evidence Audit Team (Techlaw, Inc.)
- ° Approval of drilling sub-contractor U.S. EPA Region V RPM
U.S. EPA Region V RPO

4.3 FIELD OPERATIONS

- ° Sample collection and field measurements Donohue SM and
U.S. EPA Region V RPM
- ° On-site audits Donohue TSQAM
U.S. EPA Central District Office (CDO) and/or U.S. EPA Central Regional Laboratory (CRL)

4.4 LABORATORY OPERATIONS

- ° Initiation of RAS request to RSCC Donohue authorized requester(s)
- ° U.S. EPA Region V contract administration of CLP RAS U.S. EPA Region V CRL LSSS
U.S. EPA HQ OERR
U.S. EPA Sample Management Office
U.S. EPA EMSL - Las Vegas
U.S. EPA Region V CRL LSSS
- ° Data assessment of CLP RAS data including tentatively identified compounds U.S. EPA Region V CRL LSSS and
Donohue CLP Coordinator/Data Validator
- ° Initiation of SAS request to RSCC Donohue authorized requester(s)
- ° Preparation of SAS request forms Donohue CLP Coordinator/Data Validator
- ° Contact for CLP RAS/SAS services U.S. EPA Region V CRL LSSS, RSCC

- ° Geotechnical Analysis of soil samples (grain size, Atterberg Limits, organic carbon, permeability, consolidation, triaxial shear) CLP SAS Laboratory
- ° Review and approval of CLP SAS U.S. EPA Region V CRL LSSS
U.S. EPA Region V Quality Assurance Officer
U.S. EPA Region V RPM

4.5 FIELD MEASUREMENTS

Donohue is responsible for documenting calibration and maintenance of field equipment, including pH and conductivity meters, dissolved oxygen meter, HNu, radioactivity survey meter, electromagnetic survey (EM31) meter, thermometers, and staff gauges. Donohue will also be responsible for documenting calibration and maintenance of the sampling pumps and field methane/hydrogen sulfide meter used to perform the waste mass gas investigation.

Final review and approval of field measurement and calibration procedures as described in the QAPP Appendices D and E are provided by the U.S. EPA Region V QA Officer, U.S. EPA CRL Director, and U.S. EPA Region V RPM. U.S. EPA Region V CRL and/or CDO is responsible for any identified performance and systems audits of field operations.

5.0 QUALITY ASSURANCE OBJECTIVES

5.1 INTRODUCTION

The overall Quality Assurance (QA) objective is to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide a design that is legally defensive in a court of law. Specific procedures to be used for sampling, chain of custody, calibration of field instruments, laboratory analysis, reporting, internal quality control, audits, preventive maintenance, and corrective actions are described in other sections of this Quality Assurance Project Plan (QAPP). The purpose of this section is to address the specific objectives of accuracy, precision, completeness, representativeness, and comparability.

5.2 FIELD QA SAMPLES

5.2.1 Bottle Blanks

The purpose of bottle blank samples is to assess the cleanliness of sample containers used in collecting field samples. Donohue proposes to use the bottle preparation protocols as contained in Appendix C. Bottles will be obtained from a vendor with previous participation as a bottle supplier to the National CLP Bottle Repository. Results of each case of bottles tested will be kept in the Donohue project file. In order for the case of bottles to be acceptable, test results must indicate no compounds are present above the method detection limit. Bottle blanks for water quality parameters will be assessed by filling one bottle of each lot with distilled water. The filled bottles will then be submitted to the CLP SAS laboratories labelled as a field sample. Bottle blanks for geotechnical analytes are not applicable.

5.2.2 Trip Blanks

Trip blanks will be collected to assess if any volatile organic contamination occurs during aqueous sample packaging and shipping. Trip blanks will be prepared by Donohue at their field supply storage facility by filling two 40 ml volatile vials completely with organic-free deionized water and shipped with the empty sample bottles to the field. The vials will remain capped during the sampling event and will accompany the aqueous samples collected and shipped for analysis. If detectable volatile organics are reported in the trip blanks, data will be assessed based on the criteria contained in Section IV of the "Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses," U.S. EPA, February 1, 1988.

5.2.3 Field Blanks

Field blank samples will be collected at a frequency of one per 10 or fewer aqueous samples and analyzed to check for procedural contamination from sampling equipment and/or ambient site conditions. Field blanks are collected by pouring distilled water through the decontaminated sampling equipment (bailer, bucket, pond sampler) into the appropriate sample bottles. Field blanks associated with field filtered groundwater analyzed for TAL total metals will also be filtered through a 0.45 micron filter. Assessment of the impact of detectable concentrations on site sample results will be done in accordance with Section IV of the "Laboratory Data Validation Functional Guidelines for Evaluating Organics and Inorganics Analyses," U.S. EPA February 1, 1988 and July 1, 1988.

5.2.4 Field Duplicates

Field duplicate samples are collected at a frequency of one per 10 or fewer site samples and analyzed to assess the variability of sample collection techniques and sample homogeneity. Both field and lab precision are assessed by field duplicate results, and greater relative percent difference (RPD) than lab duplicates is expected. No specific EPA criteria exist for field duplicate comparability, and data are not invalidated based on field precision.

5.2.5 Background Samples

Background samples will be collected beyond the Himco Dump Site boundaries to determine upgradient groundwater quality and native soil gas characteristics. Five existing upgradient wells will be sampled as well as three newly installed upgradient wells for a total of 27 percent of the monitoring network. This number of wells is needed to statistically determine the upgradient groundwater quality, since past data indicated variable upgradient contamination that may be from other sources.

An off-site wetland location where no known volatile organic contamination or waste disposal has occurred will be sampled for soil gas to determine if any background naturally occurring levels of methane, hydrogen sulfide, or volatile organics exist.

5.3 LABORATORY QC SAMPLES

Chemical analyses will be performed using the organic and inorganic CLP RAS analytical procedures and SAS procedures in Appendix A. The levels of laboratory QC effort for CLP RAS procedures are specified in the applicable Statements of Work (SOWs) for organics, February 1988, with revisions dated September 1988 and April 1989, and inorganics, July 1988. Specific laboratory QC samples for SAS parameters are included in each SAS request in Appendix A.

The QA objectives for precision, accuracy, and sensitivity are to achieve the QC acceptance criteria for the proposed analytical procedures. For the organic and inorganic procedures, the precision and accuracy guideline requirements are specified in the Statements of Work (SOWs). For the SAS procedures, the precision and accuracy requirements are provided in Appendix A. Specific laboratory QC measures cited in the SOWs are discussed in Sections 5.3.1 - 5.3.4 below.

5.3.1 Surrogate/Matrix Spikes - Organics

Because of the large number of organic parameters and potential matrices, it is difficult to develop precision and accuracy objectives and control limits for every organic parameter in every matrix. Therefore, it is necessary to extrapolate this information from a more limited set of compounds. This is typically done with (1) surrogate spike compounds which are added to every sample before extraction and analysis, and (2) matrix spike and matrix spike duplicate compounds which are added to selected samples before extraction and analysis. Although the surrogate and matrix spike analyses do not provide statistically valid statements about precision and accuracy for every compound in a sample, they do give the data reviewer enough information to make qualitative judgments about precision and accuracy on a sample-by-sample basis. The recoveries of the surrogate and matrix spike compounds can also be used to accept or reject data. The recoveries of the surrogate are also used as criteria to re-analyze the sample or series of samples in accordance with SOW Section 4.3.2.

5.3.2 Lab Duplicates - Inorganics

For a duplicate sample analysis, at least one duplicate sample will be analyzed from each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) for each batch of samples or for each 20 samples received, whichever is more frequent. Samples identified as field blanks can NOT be used for duplicate sample analyses. If two analytical methods are used to obtain the reported values for the same element for a batch of samples (i.e., ICP, GFAA), duplicate samples will be run by each method used. The relative percent difference (RPD) for each component will be calculated for use during data assessment.

The results of the duplicate sample analyses will be reported. An advisory limit of $\pm 20\%$ for RPD will be used for sample values greater than five times the contract required detection level (CRDL). A control limit of \pm the CRDL will be used for sample values less than five times the CRDL, and this control limit (\pm CRDL) will be entered in the "Control Limit." If one result is above the $5 \times$ CRDL level and the other is below, the \pm CRDL criteria will be used. If either sample value is less than the CRDL, the RPD is not calculated and is indicated as "NC".

5.3.3 Field Blanks

For field blank analysis, at least one field blank sample will be collected and analyzed for each group of parameters of a similar matrix. If two analytical methods are used to obtain the reported values for the same element for a batch of samples (i.e., ICP, GFAA), field blank samples will be run by each method used. The concentration for each component will be calculated for use during data assessment.

5.3.4 Matrix Spikes - Inorganics

The matrix spiked sample analysis is designated to provide information about the effect of the sample matrix on the digestion and measurement methodology. The spike is added before the digestion and prior to any distillation steps. At least one spiked sample analysis will be performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) for each batch of samples or for each 20 samples received, whichever is more frequent. Samples identified as field blanks can NOT be used for spiked sample analysis. If two analytical methods are used to obtain the reported values for the same element for a batch of samples (ICP, flame AA) spiked samples will be run by each method used. If GFAA is used, each sample is analyzed in triplicate which includes a duplicate and matrix spike. Matrix spike analysis of each sample is necessary to assess if any matrix effects are present. If the spike recovery is not within the limits of 75% to 125%, the data of all samples received associated with that spiked sample will be flagged with the letter "N". An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such a case, the spike recovery should not be considered, and the data shall be reported unflagged even if the percent recovery does not meet the 75% to 125% recovery criteria. In the instance where there is more than one spiked sample per matrix per batch, if one spike sample recovery is not within contract criteria, all samples of the same matrix in that batch will be flagged. The individual component percent recoveries (%R) will be calculated and reported.

5.4 FIELD MEASUREMENT AUDITS

The accuracy of field measurements of pH, specific conductance, temperature, dissolved oxygen, methane, hydrogen sulfide, volatile organics by HNu, electromagnetic conductivity, and radioactivity will be addressed through pre-measurement calibrations and post-measurement verifications in the field. Specifics of calibration of these instruments are contained in Appendix D and the procedure to be used to conduct the measurements are contained in the SOP in Appendix E.

The pH accuracy will be assessed by performing two measurements on three standard buffer solutions. Each measurement will be within $\pm 0.05\%$ standard unit of the certified value for the buffer solutions. Precision will be assessed through replicate measurements on field samples. The standard deviation of four replicate measurements must be less than or equal to 0.1 standard unit. The electrode will be withdrawn, rinsed with deionized water, and re-immersed between each replicates. Calibration and verification will be done in the field before the first replicate and after the last. The instrument used will be capable of providing measurements of 0.01 standard unit.

Specific conductance will be measured using a conductivity meter. The meter will be read to the nearest 10 umhos/cm within a range of 0 to 20,000 umhos/cm. Accuracy of measurements shall be ± 5 percent of a standard. Precision shall be a standard deviation of ± 10 percent.

Temperature will be measured using a thermometer with a range of -2° to 50°C and with divisions of 1.0°C . Accuracy of measurement will be $\pm 0.5^{\circ}\text{C}$. The thermometer will be calibrated against an ASTM thermometer.

Dissolved oxygen accuracy and precision will be accomplished by the water saturated air calibration procedure recommended by the manufacturer. The accuracy is expected to be ± 0.2 mg/l at the calibration temperature. Duplicate analyses are expected to agree within ± 0.1 mg/l.

Methane (CH_4) and hydrogen sulfide (H_2S) analyses will be accomplished through use of a continuous monitoring device. Calibration using a known concentration of CH_4 and H_2S will be done each day before use. A background reading will also be taken.

The HNu used for field screening volatile organic concentrations will be calibrated each day before use according to the manufacturer's specifications using a certified calibration gas containing 35 ppm isobutylene. The HNu should be calibrated to read 35 ppm at a span setting of 9.8 (10.2 ev lamp). Background readings will be taken and recorded daily from an off-site location.

The electromagnetic (EM-31) terrain conductivity meter precision is dependent on field conditions and is susceptible to cultural interference such as power lines, fences, metallic debris, etc. Goals are ± 2 percent for EM-31 full-scale values. A base station will be established on-site to conduct equipment calibration and function checks and to monitor equipment performance periodically during the field study.

Radioactivity meter calibration is initially performed and checked annually by the manufacturer. A portable check source may be used daily before field use. A background location will also be monitored daily for gross alpha, beta, gamma and X-ray radiation.

The pressure transducer used in the slug test will be set to the correct depth using the data logger. Data logger accuracy will be checked using an internal troubleshooting program.

The staff gauge increments will be verified against a reference rule and all measurements will be read from a consistent location on the bank.

Personal sampling pumps used to collect waste mass gas samples will be calibrated before each day's use against a soap bubble flow meter.

5.5 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSES

All samples will be analyzed using the CLP. Residential well samples will be analyzed by the CLP or CRL. CLP analysis of residential well samples will be done through a standardized SAS protocol developed by Region V. CRL analysis will be done using the CRL methods and Quality Assurance program. The level of QA effort for the CLP RAS analyses are specified in the CLP SOWs. In addition to routine CLP organic and inorganic analyses, SAS will be used to analyze samples for water quality parameters. These parameters and their respective QA objectives are contained in Appendix A. Soil samples will be analyzed by a SAS laboratory for physical tests by the ASTM methods contained in Appendix A. Sorbent tube samples from waste mass gas sampling will be analyzed by the SAS program for volatile organics.

5.6 COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY OF ANALYSES

5.6.1 Completeness

Completeness is defined as a measure of the number of samples actually collected compared to the number of samples required for characterization of an environmental condition and/or the amount of valid data obtained from the measurements system compared with the amount of data that was expected under normal conditions. This QA criterion is expressed in percentage. The completeness for sample collection will be 95 percent or better. The data completeness will be 100 percent for residential well samples, and 95 percent or better for other samples.

5.6.2 Representativeness

Representativeness is a measurement of the degree to which the data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, or an environmental condition. Representativeness is a qualitative criterion which is associated with the proper design of the sampling and analysis program. The data highly representative of this site will be achieved by performing all field sampling and measurements and laboratory analysis in a standardized manner and strictly adhering to the procedures specified in this QAPP, the Field Sampling Plan, and the Work Plan.

5.6.3 Comparability

Comparability is a qualitative criterion measuring the confidence with which one set of data can be compared with another. For this project, the data comparability will be achieved by the following:

- a) Analytical results will be reported in appropriate units.
- b) Same or similar sampling procedures used in the E&E 1984 field investigation will be used.
- c) Same or equivalent analytical procedures used for the E&E 1984 investigation will be used.
- d) Similar quality assurance and quality control requirements will be observed, since the CLP program will be used as it was in the E&E 1984 study.

6.0 SAMPLING PROCEDURES

The media to be sampled in the RI include landfill cap soil, landfill waste, waste mass gas, leachate, groundwater, residential well water, soil, surface water, and sediment. A complete description of sampling procedures is provided in the Field Sampling Plan (Volume 2).

7.0 SAMPLE CUSTODY PROCEDURES

7.1 INTRODUCTION

It is Donohue policy to follow the U.S. EPA Region V sample custody, and chain-of-custody protocols as described in "NEIC Policies and Procedures," EPA-330/9-78-001-R, revised June 1985. This custody is in three parts: sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under custody if it:

- * is in the possession of the sampler/analyst;
- * is in the view, after being in the possession of the sampler/analyst;
- * is in the possession of and then placed in a secured location; or
- * is in a designated secure area.

7.2 FIELD-SPECIFIC CUSTODY PROCEDURES

Documentation to be used for field collection and field sample tracking procedures are contained in each matrix specific documentation section in the Field Sampling Plan. The sample packaging and shipment procedures summarized below will insure that samples will arrive at the laboratory with the chain of custody intact.

Field procedures are as follows:

- (a) The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.
- (b) All bottles will be tagged with sample numbers and locations. The Sample Management Office (SMO) number and stickers will be affixed.
- (c) Sample tags as in Figure 7-1 are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation will explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.
- (d) The Donohue Site Manager must review all field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required. The Site Manager should notify the U.S. EPA RPM of a breach or irregularity in chain-of-custody procedures.

Figure 7-1
Sample tag and Custody Seal

Each cooler should have 2 COC seals applied.

U.S ENVIRONMENTAL PROTECTION AGENCY REGION V OFFICIAL SEAL No. 13400

Sample Tag

Project Code	Station No.	Month/Day/Year	Time	Designator: Comp. Grab	
Tag No. 5-10502	Lab Sample No.		Station Location		
	Samplers (Signatures)			ANALYSES	
Residue for: <div style="display: flex; justify-content: space-between;"> <div> Pesticides Herbicides Fungicides Insecticides Bactericides Antibiotics Priority Pollutants Organic Chemicals Inorganic Chemicals Metals Oil and Grease Nutrients Other </div> <div> <input type="checkbox"/> Yes <input type="checkbox"/> No </div> </div>			Preservative: <input type="checkbox"/> Yes <input type="checkbox"/> No		

EPA 778-312

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY REGION 5 230 South Dearborn Street Chicago, Illinois 60604

Transfer of custody and shipment procedures are as follows:

- (a) Samples are accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form (Figure 7-2). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to the RAS and SAS laboratory, or to/from a secure storage area.
- (b) Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and EPA custody seals (Figure 7-1) for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.
- (c) When samples are split with a source or government agency, a separate sample receipt (Figure 7-3) is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.
- (d) All shipments will be accompanied by the chain-of-custody form identifying the contents. The original record will accompany the shipment, the pink copy goes to SMO, and the yellow copy will be sent to the RSCC.
- (e) If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody forms as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

7.3 LABORATORY CUSTODY PROCEDURES

The chain-of-custody procedures for the CLP are described in the February 1988 and July 1988 SOWs. This same custody procedure applies to the chemical analysis of samples through the CLP SAS mechanism.

FIGURE 7-2

CHAIN OF CUSTODY FORM

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 5
230 South Dearborn Street
Chicago, Illinois 60604

CHAIN OF CUSTODY RECORD

PROJ. NO.		PROJECT NAME				NO. OF CON- TAINERS							REMARKS
SAMPLERS: (signature)													
STA. NO.	DATE	TIME	COMP.	ERAB	STATION LOCATION								
Relinquished by: (signature)		Date/Time		Received by: (signature)		Relinquished by: (signature)		Date/Time		Received by: (signature)			
Relinquished by: (signature)		Date/Time		Received by: (signature)		Relinquished by: (signature)		Date/Time		Received by: (signature)			
Relinquished by: (signature)		Date/Time		Received for Laboratory by: (signature)		Date/Time		Remarks					

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
Building 53, Box 25227, Denver Federal Center
Denver, Colorado 80225

FIGURE 7-3

RECEIPT FOR SAMPLES AND DOCUMENTS

Name of Facility	PROJ. NO.
Facility Location	

RECEIPT OF THE DOCUMENT(S) AND/OR SAMPLE(S) DESCRIBED IS HEREBY ACKNOWLEDGED			
NO.	DESCRIPTION		
Transferred by (Signature)		RECIPIENT SIGNATURE	
NAME		NAME	
TITLE	DATE SIGNED	TITLE	DATE SIGNED

7.4 FINAL EVIDENCE FILES CUSTODY PROCEDURES

Donohue maintains RI files along with all relevant records, reports, logs, field notebooks, pictures, subcontractor reports, bottle testing results, and the Laboratory Scientific Support Section (LSSS) of CRL's data reviews in a secured, limited-access area and under custody of the Donohue Site Manager.

The final evidence files from the CLP are maintained by Region V CRL LSSS Data Coordinator.

8.0 CALIBRATION PROCEDURES AND FREQUENCY

8.1 FIELD INSTRUMENTS

Field maintenance and proper documentation are important parts of the monitoring program. Before mobilization to the field, all equipment must be checked for malfunctions and cleaned. In addition, all meters must be calibrated by the sampling team on-site to ensure proper working order and to render integrity to the measured values. Calibration procedures provided by the manufacturer will be followed for the HNu, pH meter, conductivity meter, dissolved oxygen meter, radiological survey meter, and gas monitor. These procedures are attached as Appendix D.

The Site Quality Control Officer is responsible for maintaining the field meter calibration log for field instruments (Appendix A of the FSP). This log contains the same information as that for permanent lab instruments (EPA ID number, name, and model of meter). The log also contains calibration results and notes for each day of equipment use. Documentation shall include:

- ° Date of calibration.
- ° Initials of person performing calibration.
- ° Calibration results.

8.2 LABORATORY INSTRUMENTS

Laboratory calibration procedures and frequency will be done in accordance with each laboratory SOW and the individual CLP SAS requests in Appendix A.

9.0 ANALYTICAL PROCEDURES

Analytical procedures to be used for the Himco Dump RI are:

- ° CLP RAS inorganics and organics methods for all enforcement, litigation, and evidentiary data as contained in SOW-February 1988 and SOW-July 1988.
- ° SAS methods supplied by EPA Region V for physical soil tests (grain size, Atterberg limits), residential well analysis, organic carbon in soil, sulfate, chloride, nitrate and nitrite, chemical oxygen demand, total phosphorus, alkalinity, total suspended solids, total dissolved solids, total Kjeldahl nitrogen, ammonia, and volatile organics in sorbent tubes.
- ° USGS procedure for bromide as contained in Appendix A - SAS Request prepared by Donohue.
- ° ASTM analytical procedures for physical soil tests (permeability, consolidation, triaxial shear) as contained in Appendix A - SAS Request prepared by Donohue.

9.1 ROUTINE ANALYTICAL SERVICES (RAS) LABORATORY PROCEDURES

The current EPA CLP Statement of Work (SOW) for Organics, February 1988, with revisions dated September 1988 and April 1989 and the SOW for Inorganics Analysis, July 1988, specify the analytical procedures to be used. Included in the SOW are sample custody procedures, instrument calibration procedures, and frequency of calibration. Due to the expected percent levels of calcium expected in the landfill cap soil samples, calcium data obtained through the CLP inorganic SOW for these samples may not be valid.

9.2 SPECIAL ANALYTICAL SERVICES (SAS) LABORATORY PROCEDURES

The analytical procedures to be used for performing the SAS analyses are specified in each SAS request in Appendix A. Specified in the SAS requests are calibration procedures, frequency of calibration, and the internal quality control checks required for each analysis.

9.3 FIELD SCREENING ANALYTICAL PROCEDURES

The procedures for field measurements are described in the SOPs contained in Appendix E. Field measurement of groundwater, surface water, and leachate for pH, conductivity, dissolved oxygen, and temperature will be done. Field measurement of waste mass gas methane and hydrogen sulfide concentrations will be performed during the RI. Screening of soil samples for volatile organics by HNu and radioactivity will also be conducted.

10.0 INTERNAL QUALITY CONTROL CHECKS

10.1 RAS INTERNAL QUALITY CONTROL CHECKS

Internal quality control procedures for RAS from the CLP are specified in the SOWs for organics and inorganics as well as in the methods descriptions. These specifications include the types of QC checks required (method blanks, reagent/preparation blanks, matrix spike and matrix spike duplicates, calibration standards, internal standards, surrogate standards, the frequency of each audit, the specific calibration check standards, laboratory duplicate/repligate analysis), compounds and concentrations to be used, and the quality control acceptance criteria for these audits.

10.2 SAS INTERNAL QUALITY CONTROL CHECKS

Quality control checks for SAS are identified in the SAS requests in Appendix A.

10.3 FIELD MEASUREMENT

QC procedures for field measurements are limited to checking the reproducibility of the measurement by obtaining multiple readings on a single sample or standard and by calibrating the instruments. Assessment of field sampling precision and bias will be made through collection of field duplicates and field blanks in accordance with the applicable procedures described in the FSP at the frequency indicated in the Sampling and Analysis Summary (Table 3-5).

10.4 INTERNAL AUDIT PROCEDURE

The TSQAM is responsible for planning, scheduling, and coordinating evaluations by the Donohue Technical Advisory Committee (TAC) of internal QC checks performed in accordance with the QAPP. The TAC will evaluate the procedures performed by field personnel in accordance with the FSP and QAPP. This evaluation will include review of the following:

- ° Possession and use of the latest approved procedure(s), standards, and/or project-specific instruction(s).
- ° Conformance with appropriate procedures, plans, standards, and instructions.
- ° Identification, accuracy, and completeness of paperwork generated during performance.

11.0 DATA REDUCTION, VALIDATION, AND REPORTING

11.1 DATA REDUCTION

RAS organic and inorganic results will be reported using the Organic Analysis Data Sheets (OADS) and Inorganic Analysis Data Sheets (IADS) used by the CLP. The data package will contain all deliverables listed in the CLP SOWs. Soil values will be reported on a dry weight basis. SAS analyses will be reported in accordance with the SAS deliverables listed on separate forms, to include EPA sample number and description, date of analysis, analyte(s), detection limit(s) in the sample matrix analyzed, appropriate units, and data flags appropriate to the analyses. RAS and SAS result forms and data packages will be submitted to EPA Region V LSSS.

Following validation by EPA of the RAS and SAS data packages, reduced data will be forwarded to the Donohue Site Manager along with the EPA data review. The data will include the analytical results reported and any data flags provided by the laboratory and data reviewer. Data flags may include: (1) estimated concentration due to poor spike recovery, (2) concentration of chemical also found in lab blank, and (3) concentration below required detection limit. Data qualifier codes specified in the CLP SOW may also be used. Reviewer comments may include: (1) usable as a quantitative result, (2) usable with caution as an estimated concentration, and (3) unusable due to out-of-control QC results.

11.2 DATA VALIDATION

Validation will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements contained in RAS and SAS methods. Raw data such as GC/MS chromatograms and mass spectra, ICP and GFAA data reports, and data station printouts will be examined to ensure that reported results are accurate. The EPA LSSS will be responsible for this. The protocol for RAS analyte data validation as presented in "Functional Guidelines for Evaluation Organic and Inorganic Analyses" will be used.

11.3 DATA REPORTING

11.3.1 RAS Data

The RAS analytical laboratories will prepare and submit full analytical and QC reports to EPA Region V in compliance with requirements of the CLP to include the following (as applicable):

1. Narrative including statement of samples received, description of any deviations from RAS standard procedures, explanation of qualifications regarding data quality, and any other significant problems encountered during analysis.

2. Up to 20 extractable organic compounds not included in the RAS analytes, tentatively identified and quantified against the nearest internal standard.
3. An organic QA/QC report including Forms I to X, surrogate spike results for each sample, matrix spike, and matrix spike duplicate results, method blank results, initial and continuing calibration checks.
4. An inorganic QA/QC report including Forms I to XIII, spike and duplicate results, method blank results, and initial and continuing calibration checks.
5. Field and laboratory chain-of-custody documentation pertaining to each sample delivery group analyzed.

11.3.2 SAS Data

The SAS analytical laboratories will prepare and submit data packages to EPA Region V in compliance with the specific deliverables listed in Section 9 of the SAS request in Appendix A which include the following (as applicable):

1. Narrative including a listing of samples received with test procedure reference (ASTM or EPA) listed.
2. All handwritten raw data, computer spreadsheets, and instrument printouts sufficient to allow independent calculation of results reported.
3. All calibration curves, bench records of reagent standardization, results of laboratory blanks, duplicates, matrix spikes, EPA QC reference samples with true values and associated 95 percent confidence limits.
4. Field and laboratory chain-of-custody documentation pertaining to each case of samples analyzed.

12.0 PERFORMANCE AND SYSTEMS AUDITS

12.1 INTERNAL AUDITS

Systems audits of on-site field collection activities will be conducted. The audits will include verification that approved procedures are in place and used, that an acceptable calibration program is operational, that an organizational structure is in place and personnel responsibilities are clearly defined, that a training program for personnel is documented and current, that the chain-of-custody program and records retention program are adequate, and that corrective actions are taken by field personnel in a responsive and timely manner.

Before field activities start, and/or shortly after systems are operational, the Technical Advisory Committee (TAC) will conduct a field sampling system audit on-site to review:

- ° Organization and responsibilities in order to determine whether the field team organization is operational.
- ° The collection of samples to assure that written procedures are available and are being followed.
- ° Chain-of-custody program to assure that the appropriate steps have been followed in the traceability of samples.
- ° The implementation of the operational procedures to assure that the appropriate QC checks are being made in the field and records are maintained of these checks.

12.2 EXTERNAL AUDITS

The CLP RAS and SAS laboratory facilities participate regularly in the performance evaluation programs administered by EPA. U.S. EPA EMSL - Las Vegas conducts annual system audits of the CLP laboratories and sends quarterly performance evaluation samples for ongoing assessment of laboratory precision and accuracy. The U.S. EPA Region V CRL is responsible for auditing laboratories performing SAS analyses. The field activities may be audited by the EPA Region V CDO and/or CRL at the request of the RPM.

13.0 PREVENTIVE MAINTENANCE

13.1 CLP RAS AND SAS LABORATORIES

All CLP laboratories are required under Exhibit E QA/QC Requirements in the organics and inorganics SOW to have Standard Operating Procedures (SOPs) for preventive maintenance of each instrument measurement system. All maintenance activity is documented on standard forms maintained in logbooks. A history of the maintenance records of each system serves as an indication of the adequacy of maintenance schedules and parts inventory.

13.2 FIELD MEASUREMENTS

Specific preventive maintenance procedures to be followed for field equipment are those recommended by the manufacturer and are contained in the Field Sampling Plan (Appendix C) and QAPP Appendix E.

Field instruments will be checked and calibrated daily before use. Calibration checks will be documented on the field meter instrument calibration logs (FSP, Appendix A). Critical spare parts such as batteries will be kept on-site to reduce down time. Backup instruments and equipment should be available on-site or within one-day shipment to avoid delays in the field schedule.

**14.0 SPECIFIC ROUTINE PROCEDURES TO ASSESS
DATA PRECISION, ACCURACY,
AND COMPLETENESS**

14.1 FIELD DATA

Data assessment will be accomplished by the Site QC Officer (SQCO). The SQCO will review field results for compliance with the established QC criteria as described in the QAPP and FSP. Field data will be assessed by examination of field meter daily calibration accuracy, field duplicate, and field and back-ground blank contents. Field duplicate precision will be expressed based on the relative percent difference (RPD) statistic as described in the CLP SOWs. Any problems arising during sample collection, packing, shipping, or analysis will be taken into consideration.

14.2 LABORATORY DATA

Completeness of RAS data based on contractual completeness is assessed before release to Region V by the SMO using the Contract Compliance Screening Procedure. RAS data will then be reviewed for precision and accuracy by Region V LSSS in accordance with the U.S. EPA Hazardous Site Evaluation Division, "Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses," "Laboratory Data Validation Functional Guidelines for Evaluating Pesticides/PCBs Analyses," and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses."

Analytical data for SAS analysis will be assessed by the SMO for completeness based on the SAS request requirements. Precision and accuracy of SAS analytes will be assessed by EPA Region V LSSS by a review of the data package and QA/QC results to determine if the protocols specified in the SAS request were adhered to.

15.0 CORRECTIVE ACTION

15.1 DISCOVERY OF QA PROBLEMS

The TAC will issue a description of each nonconforming condition identified, i.e., when objectives for field precision, accuracy, completeness, representativeness, or comparability are not satisfied, or when unacceptable procedural practices or conditions are identified during ongoing review or system audits.

A full description will include the conditions requiring corrective action, indicate the nature of the corrections required, and specify a schedule and the authority for correction.

15.2 CORRECTIVE ACTION

Upon the documentation of a QA problem, it will be relayed to the responsible officer of the laboratory, the Project Manager, the Site Manager, Field Team Leader, and Site QC Officer. The responsible individual will indicate the nature of the corrective action taken and will require appropriate documentation of such action. The corrective action taken will include measures to prevent a recurrence.

15.3 STOP-WORK ORDER

If corrective actions are insufficient, resolution cannot be reached, or results or prior work are indeterminate, work may be stopped by the TSQAM. If there is a disagreement between the TSQAM and the Site Manager as to the stop-work directive, the differences shall be brought to the attention of the PM for resolution.

15.4 CLP LABORATORIES

The laboratories participating in the CLP are required to have a written SOP specifying corrective action to be taken when an analytical error is discovered or the analytical system is determined to be out of control. The SOP requires documentation of the corrective action and notification of the analyst of the error and correct procedures.

The SMO also may request corrective action for any contractual non-conformance identified by audit or data validations. The EPA Region V LSSS may request corrective action by the CLP laboratories for any non-conformances identified in the data validation process through the SMO or, for minor problems, the lab may be contacted directly. Corrective actions may consist of sample re-analysis if holding time permits. Recollection of samples may be necessary if holding times have expired.

15.5 SPECIAL ANALYTICAL SERVICES

Corrective action for SAS requests should comply with the specific SAS request as well as with the procedures described in Section 15.4.

15.6 FIELD CHANGES

The Site Manager is responsible for all site activities. In this role, the Site Manager at times is required to adjust the field program to accommodate site-specific needs. When it becomes necessary to modify a program, the Field Team Leader notifies the Site Manager of the anticipated change, and documents and implements the necessary changes. The EPA RPM and PO will be notified if the change is determined to be a significant one. Significant field changes may include: deleting a sampling location, using less inert sampling devices, or changing from the chemical analyses listed in the QAPP and FSP.

16.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The complete and correct implementation of the QAPP will be reviewed by the TAC and an assessment of adherence to sample collection procedures and data quality will be presented in the data quality summary report.

Meetings to discuss the progress of the project will be held as requested by EPA. Concerns which arise during the course of the work that might require significant changes to the scope of work or departures from the protocols specified in the approved project plans will be discussed at these meetings. Proposed adjustments will be submitted to the EPA in writing. At requested intervals, the TSQAM, in conjunction with the TAC, will summarize QA activities. These summaries may include:

- ° Results of system and performance audits conducted during the period.
- ° An assessment of the accuracy of measurement data, precision, completeness, representativeness, and comparability including soil physical tests.
- ° A listing of the QA problems discovered during the period, related corrective actions undertaken, and an assessment of the results of these actions.
- ° Identification of significant QA problems and recommended solutions.

APPENDIX A
SAS REQUEST FORMS

Water Quality SAS:	Chloride, sulfate, TDS, TSS, alkalinity, TP, TKN, NH ₃ , NO ₂ and NO ₃ , COD
Bromide SAS:	Anions, ion-exchange chromatographic, automated
Residential Wells Organic SAS:	Analysis of drinking water/residential wells for volatiles, semi-volatiles, and pesticides/PCBs with low quantitation limits
Residential Wells Inorganic SAS:	Analysis of drinking water/residential wells for metals and cyanide
Sorbent Tube SAS:	Analysis of sorbent tubes for TCL volatile organics
Geotech SAS:	Analysis of soil samples for Atterburg Limits, particle size analysis, permeability, consolidation, and triaxial shear
Organic Carbon SAS:	Determination of percent organic carbon in soil on air dried sample

WATER QUALITY SAS

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request



Regional Transmittal



Telephone Request

- A. EPA Region/Client: REGION V
- B. RSCC Representative: JAN PELS
- C. Telephone Number: (312) 353-2720
- D. Date of Request: _____
- E. Site Name: Himco Dump, Elkhart Indiana

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of 77
low level groundwater samples for chloride,
sulfate, TDS, TSS, alkalinity, TP, TKN, NH₃, NO₂+NO₃,
COD. Samples will be unfiltered, * analyses may require filtering
in lab. Report results in mg/l, note if filtration required,
and report as dissolved.
2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):
- 77 ^{AQUEOUS} whole samples - low concentration (see Table 1
for historical data) in 4-1 liter HDPE bottles:
2L Unpreserved (Cl, SO₄, TDS, TSS, alkalinity)
2L preserved w/ H₂SO₄ (TP, TKN, NH₃, NO₂+NO₃, COD)
3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):

Superfund Remedial Investigation

TABLE 1

historical levels - thmco dump samples

MATRYE	Range (mg/l)	POSSIBLE INTERFERENCES
Chloride	8-98	sulfate
sulfate	5 - 620 *	high solids
TDS	222-7830 *	high solids
TSS	unknown	high solids - turbidity
alkalinity	150 - 2380 *	sulfide
TP ¹	unknown	
TKN ²	unknown	
NH ₃	0.02 - 450 *	
NO ₂ + NO ₃	< 0.005 - 3.8	
COD	unknown	
1	historical dissolved orthophosphate levels	< 0.005 - 0.60 mg/l
2	historical dissolved organic nitrogen levels	< 0.005 - 750 mg/l
* well M-1		

TABLE 2 HOLDING TIMES - HINCO DUMP

F NAUITE	
Chloride	28 days
sulfate	28 days
TDS	7 days
TSS	7 days
alkalinity	14 days
TP	28 days
TN	28 days
NH ₃	28 days
NO ₂ + NO ₃	28 days
COD	28 days

4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: _____
_____ daily by overnight carrier _____
6. Number of days analysis and data required after laboratory receipt of samples: _____
_____ 30 days _____
7. Analytical protocol required (attach copy if other than a protocol currently used in this program): _____
_____ see specific sheets attached for each method _____
8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): _____ total _____
_____ holding times should not exceed those listed in Table 2 _____
_____ (from date of collection (field) to analysis). call _____
_____ contact if times^{was} exceeded prior to lab _____
_____ receipt, so recollection can occur. _____
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion. _____
_____ see specific sheets attached _____
10. Other (use additional sheets or attach supplementary information, as needed): _____
11. Name of sampling/shipping contact: _____ Marcia Kuehl _____
Phone: _____ (414) 458-8711 x 2253 _____

4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: daily by overnite carrier
6. Number of days analysis and data required after laboratory receipt of samples: 30
7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
1. EPA Method 325.1 (Colorimetric, Automated Ferricyanide, AA-I) 1983ed., or
 2. EPA Method 325.2 (Colorimetric, Automated Ferricyanide, AA-II) 1983ed., or
Note: A Region V CRL Auto Analyzer Manifold is attached for Method 325.2 to correct errors in Method 325.2's manifold diagram.
 3. ASTM Colorimetric Method (Manual Method) -ASTM D 512C-81, or
 4. Method 407C (Potentiometric Titration) Standard Methods, 16th ed. Samples will be kept at 4°C until analysis and validation of results.
8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
- For colorimetric methods (1) use a standard curve between 0 and 300 mg/l or less, (2) the calibration curve must include 5 points or more (including a zero concentration standard), and (3) samples with absorbances or peak heights greater than highest standard must be diluted and reanalyzed. For titrimetric method 1) use either 0.0141 or 0.025 N titrant, 2) automated potentiometric titrators are acceptable, 3) do not use more than 20 ml titrant for 50 ml or 100 ml sample aliquots, 4) dilute and reanalyze any sample aliquots requiring more than 20 ml titrant, 5) remove any interfering chromate, ferric iron, sulfide, and sulfite, and 6) standardize titrants daily. Obtain approval of CPMS, CRL prior to use of any other method.
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.
- The test procedure used will be clearly identified. For the colorimetric methods, bench records tabulating order of calibration standards, verification and control standards, samples, matrix spikes, titrant blanks, etc. with resulting peak height, concentration, or absorbance read-outs will be provided with copies of worksheets used to calculate results. For the titration method, any potentiometric titration curves and all bench records tabulating titrant standardization, samples, aliquot volumes, matrix spikes, etc. will be provided. Records of titrant standardization and titrant blanks will be provided. A photocopy of instrument readouts, i.e. strip charts, printer tapes, etc. must be included for all analyses. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA audit results. EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.
10. Other (use additional sheets or attach supplementary information, as needed): _____
11. Name of sampling/shipping contact: Marcia Kuehl
- Phone: 414 458 8711 x2253

3.

1. DATA REQUIREMENTS

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired (+% or Conc.)</u>
<u>Chloride</u>	<u>5 mg/l</u>	<u>Differences in duplicate sample results are to be <5 mg/l for concentrations <50 mg/l and are to be < 10% for concentrations exceeding 50 mg/l. The significant figures to report depend on sen- sitivity of colorimetric curve or number of signifi- cant figures in titrant volume.</u>
<u>Note: These are minimum requirements</u>		
<u>Report actual detection limit used,</u>		
<u>based on allowable methodology options.</u>		

II. QC REQUIREMENTS - Do not use designated field blanks for QA Audits.

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
a) For Methods 325.1, 325.2, and ASTM D 512C		
<u>Matrix Spike*</u>	<u>1 per group of 10 or fewer samples</u>	<u>85 - 115% Recovery</u>
<u>Lab Duplicate</u>	<u>" "</u>	<u>+ (10% or 5 mg/l)</u>
<u>Lab Blank</u>	<u>" "</u>	<u><5 mg/l</u>
<u>Calibration Verification Std.</u>	<u>" "</u>	<u>90-110% Recovery</u>
<u>1 Set of EPA QC Mineral Ref. Samples - 2 Concentrates</u>	<u>1 per sample set</u>	<u>85-115% Recovery</u>
b) For Method 407C		
<u>Same as Item IIa for Matrix Spike*, Lab Duplicate, and QC Mineral Reference Samples.</u>	<u>Beginning and end of sample set</u>	<u>-3 to +3 mg/l</u>
<u>Lab Blank (Not Titration Blank)</u>	<u>At end of sample set</u>	<u>95 - 105% Recovery</u>
<u>Calibration verification Standard (Same as Titrant Standardization)</u>		

*Matrix spike concentrations will be greater than 30% of the sample concentration, but spiked sample shall not exceed the working range of the standard curve or titration.

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact SMO:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

SO4-1

- 2 -

4. Estimated date(s) of collection: _____
- Estimated date(s) and method of shipment: daily by overnight carrier
6. Number of days analysis and data required after laboratory receipt of samples: 30
7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
1. EPA Method 375.2 (Colorimetric Methylthmol Blue) - 1983 ed.
- Note: This method requires 0.75 mg/l SO₄ in Dilution Water (See Reagent Section 6.8)
 2. Method 426C of Standard Methods, 16th ed. (Turbidimetric)
- Note: this last method provides for measurement of sulfate using 2 standard curves - 1 for sulfate concentrations between 0 and 10mg/l, and 1 between 10 and 40 mg/l sulfate.
- Samples will be kept at 4°C until validation of results.
8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
- Sample holding time is not to exceed 28 days from date of sample collection. Sulfate standards will be prepared daily from stock solution. Samples with absorbances or turbidities greater than that in the highest standard will be diluted and rerun. For Method 426C, 1) the reanalysis solution should contain between 20 and 40 mg/l sulfate, and 2) concentrations must be corrected for background turbidity and color per Section 5d of Method 426C using pH adjusted sample aliquots. Use only the methods specified. Calibration curves must include at least 6 points (including a zero concentration standard) for Method 375.2 and Buffer A of Method 426C.
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.
- The test procedure used must be clearly identified. Results shall be reported as mg/l SO₄. Bench records tabulating the order of calibration standards; lab control standards, lab blanks, samples, spikes, etc., with resulting absorbances or concentration readouts, will be provided along with copies of worksheets used to calculate results. Background absorbances used for turbidity corrections must be tabulated for each sample aliquot tested. A photocopy of the instrument readout (ie. strip charts, printer tapes, etc.) must be included. All records of analysis must be legible and sufficient to calculate all concentrations and results. EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.
10. Other (use additional sheets or attach supplementary information, as needed):
- Report which samples (if any) required filtration prior to analysis
11. Name of sampling/shipping contact: Marcia Kuehl
- Phone: 414-458-8711 x 2253

SO4-2 -3-

I. DATA REQUIREMENTS

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (% or Conc.)
<u>Sulfate</u>	<u>5 mg/l</u>	<u>Method 375.2:</u> Differences in duplicate sample results are to be < 5 mg/l for concentrations < 50 mg/l, and < 10% for concentrations > 50 mg/l.
		<u>Method 426 C:</u> Differences in duplicate sample results are to be < 2 mg/l for concentrations < 20 mg/l and < 10% for concentrations > 20 mg/l in aliquot tested.
<u>Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.</u>		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits.

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
<u>Matrix Spike*</u>	<u>1 per group of 10 or fewer samples</u>	<u>85-115%</u>
<u>Lab Duplicate</u>	<u>" "</u>	<u>+ (10% or 5 mg/l) for Method 375.2</u>
		<u>+ (10% or 2 mg/l) for Method 426C</u>
<u>Lab Blank (0 mg/l SO₄)</u>	<u>" "</u>	<u>< 5 mg/l - Method 375.2</u>
		<u>-2 to +2mg/l-Buffer B of Method 426C or</u>
<u>Lab Blank (10 mg/l SO₄)</u>	<u>" "</u>	<u>8 to 10mg/l - Buffer A of Method 426C</u>
<u>Calibration Verification Standard</u>	<u>1 per group of 10 samples and at end of sample set</u>	<u>90 - 110%</u>
<u>1 Set of EPA QC Mineral Reference Samples</u>	<u>once per sample set</u>	<u>85-115% for each concentration.</u>

*Matrix spike concentrations will be greater than 30% of sample concentrations, but spiked samples shall not exceed working range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact SMO.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

6. Approximate number of days results required after lab receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 160.1, 1983 ed., or

2. Method 209B, "Standard Methods", 16th ed. Samples will be kept at 4°C until

sample analysis and validation of results. Holding time is 7 days from date of

sample collection.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1) Use standard aliquots of 100ml;

however do not use sample aliquots yielding more than 200 mg residue. If residue is greater than 200 mg, repeat the analysis using a smaller sample aliquot. 2) If the pH value is less than 4.0, raise the pH of the aliquot (using NaOH titrant) to between pH 4 and 8 and subtract the weight of sodium added from the weight of the residue.

3) Residue will be weighed either to constant weight pursuant to Section 7.6 of Method 160.1 the final weight is to be used for calculations. Constant weight is defined as

a) less than 0.5 mg or less than 4% weight loss from the previous weight, whichever is smaller, or b) dried overnight (12 hours drying time) with a single weight used for calculations.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the QC reference sample lot numbers used and their true values with 95% confidence intervals. Bench records of tare weights, final weights, additional weights to determine constant weights, volumes filtered, blanks, duplicate samples, and reference samples will be provided with copies of work sheets used to calculate results. Dates and time of 1) determination of tare weights, 2) sample filtration, and 3) determination of residue weights and constant residue weights will be part of bench records. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Marisa Kuenl

Phone: 414-458-8711 X 2253

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

<u>Parameter</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (+% or Conc.)
TDS	20 mg/l	Difference in duplicate sample aliquots shall not exceed 2 mg for residues. Duplicate differences shall not exceed 10% for sample values greater than 200 mg/l.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		

II. QUALITY CONTROL REQUIREMENTS Do not use any designated field blanks for QA Audits.

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (+% or Conc.)</u>
1. 1 set of EPA OC Mineral Reference Samples*- 2 concentration levels.	1 per sample set	85-115% Recovery
2. Lab Duplicate	At least 1 per group of 10 or fewer samples	+ (10% or 2 mg of residue)
3. Lab Blanks (100 ml of filtered reagent water)	At least 1 per group of 10 or fewer samples	- 20 mg/l to + 20 mg/l

* Alternate reference samples must be approved by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:

Take corrective action and ^{reanalyze} ~~reject~~ samples. Contact SMO.

6. Approximate number of days results required after lab receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 160.2, 1983 ed., (Gravimetric, Dried at 103° - 105° C) using glass fiber filter discs without organic binder such as: Millipore AP-40, Reeve Angel 934-AH, Gelman A/E, or equivalent. Use only membrane filter apparatus with 47 mm diameter glass fiber filter and a coarse (40-60 micron) fritted disc filter support. The filter and support specifications are mandatory. Samples will be held at 4°C until sample analysis and validation of results are completed. Holding time is 7 days from date of sample collection.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Sample aliquot volumes are selected on the basis of the following factors. a) During initial sample filtration, filtration rate should not drop rapidly, or require more than 5 minutes of filtration time. (Increase the filter area or decrease the sample volume as needed for sample reanalysis), b) The sample aliquot filtered should provide a residue with greater than 1.0 mg for aliquots less than 200ml in volume, and c) Sample aliquots should not exceed 200ml in volume. 2. Duplicate sample aliquots will be filtered with 2 or more intervening samples. 3. Final residues are to be weighed either to constant weight pursuant to Section 7.6 of Method 160.1 (The final weight is to be used for calculations), or dried overnight (12 hours of drying time) with the single weight used for calculations. Constant weight is defined as less than 0.5 mg or less than 4% weight loss from the previous weight, whichever is smaller. 4. Use only the method specified above in items 7 and 8.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify EPA QC reference sample lot numbers used and their true values and 95% confidence intervals. Bench records of tare weights, final weights, volumes filtered, blanks, duplicate samples, and reference samples (all in the order filtered) will be provided along with copies of worksheets used to calculate results. Dates and time of a) filtration of initial 100ml volume, b) determination of tare weights, c) sample filtration, and d) determination of constant residue weights will be part of bench records. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Marcia Kuehl

Phone: 414-458-8711 x2253

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

<u>Parameter</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (+% or Conc.)
<u>Suspended Solids</u>	<u>2-3 mg/l for 200 ml</u>	<u>Difference in duplicate</u>
<u>Note: These are minimum</u>	<u>sample aliquot</u>	<u>results shall not exceed</u>
<u>requirements. Report the</u>		<u>0.5 mg for duplicate</u>
<u>actual detection limits</u>		<u>aliquots filtered.</u>
<u>used based on allowable</u>		
<u>methodology options.</u>		

II. QUALITY CONTROL REQUIREMENTS Do not use designated field blanks for QA Audits.

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (+% or Conc.)</u>
<u>1) Lab Duplicates</u>	<u>1 per group of 10 or</u>	<u>less than 0.5 mg for residue <</u>
<u>(See item 8.3 on Page 2)</u>	<u>fewer samples</u>	<u>less than 10% for sample residue</u>
		<u>> 5 mg</u>
<u>2) Lab Blanks</u>	<u>1 per group of 10 or</u>	<u>-0.5 to +0.5 mg</u>
<u>(200 ml aliquots)</u>	<u>fewer samples</u>	
<u>3) 1 set of 2 EPA OC</u>	<u>1 per sample set</u>	<u>< 5 mg/l error for con-</u>
<u>Residue Reference</u>		<u>centrations ≤ to 50 mg/l</u>
<u>Samples-2 concentration</u>		<u>or < or = to 10% for nom-</u>
<u>levels</u>		<u>inal concentrations > than</u>
		<u>50 mg/l</u>

* Alternate reference samples must be approved by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:Take corrective action and reanalyze samples.Contact SMO

4. Estimated date(s) of collection: _____
Estimated date(s) and method of shipment: Daily by overnight carrier
6. Number of days analysis and data required after laboratory receipt of samples:
Laboratory should report results within 30 days of receipt of samples.
7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
1) Alkalinity EPA Method 310.1 (Titrimetric, pH 4.5) 2) Standard Methods, 16th Edition, Method 403 4c and 4d.
Samples will be stored at 4°C until analysis and validation of results.
8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
Samples holding time should not exceed 14 days from date of collection. Use potentiometric titration to pH 4.5 for alkalinity > 20 mg/l as CaCO₃. For concentrations <20 mg/l, use EPA Method 310.1 (Section 6.3) or Standard Methods, Method 403 4d. Do not use titrant volumes greater than 50ml. Obtain approval of CPMS, CRL prior to use of any other method.
Use Na₂CO₃ to standardize titrant. Standardize the pH meter and the titrant each day.
Standardize the pH meter using at least two buffers which bracket the end point.
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:
The Test procedure used will be clearly identified. Bench records tabulating the order of analysis including pH meter calibration, titrant standardization, lab blanks, samples, lab control standards, duplicates, etc., with resulting titrant volumes or readouts will be provided along with calculation worksheets. All records will be legible and sufficient to recalculate all sample concentrations and QA audit results. Report method of titrant standardization. EPA QC Reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.
10. Other (use additional sheets or attach supplementary information, as needed): _____
11. Name of sampling/shipping contact: Marcia Kuehl
Phone: 414-458-8711 x2253

21K-2

I. DATA REQUIREMENTS

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (+% or Conc.)
<u>Alkalinity</u>	<u>2 mg/l for low level</u>	<u>+ 2 mg/l for Conc.</u>
	<u>20 mg/l for high level</u>	<u>< 20 mg/l CaCO₃</u>
		<u>+ 10% for Conc.</u>
		<u>> 20 mg/l</u>
NOTE: These are minimum requirements. Report actual detection limits used based on allowable methodologies.		

- II. QC REQUIREMENTS - Do not use designated field blanks for QA audits.
The QA audits below will be done for each group of low-level and high-level alkalinity determinations.

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
<u>lab blank</u>	<u>at least 1 per group of 10 or fewer samples</u>	<u><10 mg/l for high-level samples tested.</u> <u><2 mg/l for low-level samples tested.</u>
<u>lab duplicate</u>	<u>at least 1 per group of 10 or fewer samples</u>	<u>+ 10% or + 2 mg/l</u>
<u>lab control sample</u> <u>1 set of EPA QC mineral reference samples</u>	<u>1 per sample set</u>	<u>90-110% recovery.</u>

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: daily by overnight courier
6. Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days after receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Total Phosphorus EPA Method 365.1 (Automated, Colorimetric, Ascorbic Acid)

Total Phosphorus EPA Method 365.2 (Automated, Colorimetric, Single Reagent)

Total Phosphorus EPA Method 365.4 (Block Digestor)

Samples will be preserved in the field with 1 ml/l H₂SO₄ to pH <2 and stored at 4°C

until analysis and validation of results.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.) Check sample pH using wide-range pH paper. If the pH > 2, contact CPMS, CRL for instructions:

Dilute and redigest samples with absorbances or peak heights higher than the highest standard. All standards, blanks, audits, etc. must be digested. The holding time is not to exceed 28 days from sample collection. Use only the method(s) specified above. The calibration curve must include at least 5 standards. (One of the standards must be zero concentration).

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:

The test procedure used will be clearly identified. Bench records and all records of analysis and calculations for samples, blanks, duplicates, spikes and all control checks with peak height or response and concentrations will be provided with copies of worksheets. Results will be reported as mg/l P. Any digestion log will be provided showing sample aliquots and concentrations of all samples tested. Records must be legible and sufficient to recalculate all concentrations. A photocopy of the instrument readout i.e. stripcharts, printer tapes, etc. must be included. EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

Denote which samples (if any) required filtration prior to analysis

Name of sampling/shipping contact: Marcia Kuehl

Phone: 414-458-8711 x 2253

I. DATA REQUIREMENTS

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (+% or Conc.)
<u>Total P</u>	<u>0.05 mg/l</u>	<u>Duplicate results must agree to within 10% for concentrations > 0.5 mg/l or within 0.05 mg/l for concentrations < 0.5 mg/l</u>
<u>NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.</u>		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
<u>Matrix Spike*</u>	<u>at least 1 per group of 10 or fewer samples</u>	<u>85% - 115%</u>
<u>Lab Duplicate</u>	<u>at least 1 per group of 10 or fewer samples</u>	<u>+ (10% or 0.05 mg/l)</u>
<u>Lab Blank (Also serves as a calibration blank).</u>	<u>at least 1 per group of 10 or fewer samples</u>	<u><0.05 mg/l</u>
<u>Calibration verification standard</u>	<u>1 per group of 10 samples and end of sample set</u>	<u>90% - 110%</u>
<u>1 set of EPA nutrient QC reference samples conc. 3&4</u>	<u>1 per sample set</u>	<u>85% - 115%</u>

*The matrix spike concentrations will be approximately 30% or larger of sample concentrations, but spiked samples shall not exceed the working range of the standard curve.

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: daily by overnight courier
6. Number of days analysis and data required after laboratory receipt of samples:
Laboratories shall report results within 30 days after receipt of samples
7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
1) EPA Method 351.2 (Colorimetric, Block Digestor, AA II)
2) EPA Method 351.3 (Colorimetric, Titrimetric, or Potentiometric) (NOTE: For Method 351.3 the micro-Kjeldahl technique is not acceptable.) Samples will be preserved in the field using H₂SO₄ (1ml/L) to pH<2, samples will be stored at 4°C until analysis and validation of results.
8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
For all Methods: Analyze samples within 28 days after collection. Check the sample pH (wide range pH paper). If the pH>2, contact CPMS, CRL for instructions. Use nicotinic acid for the control standard. Use an organic nitrogen compound for the matrix spike. Use only the Methods specified in item 7. Method 351.3 requires distillation separation, prior to all final ammonia measurements. For Method 351.3: Use only the Colorimetric method for samples containing less than 1 mg N/l.
For Colorimetric Methods (351.2 and 351.3): Use at least five calibration standards (including a zero concentration standard). Dilute and reanalyze samples with concentrations that exceed the highest calibration standard.
For the Potentiometric Method (351.3): Use at least four calibration standards. Dilute and reanalyze samples with concentrations that exceed the highest calibration standard.
For the Titrimetric Method (351.3): Standardize the titrant each day. Include records of indicator blank.
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.
Identify the test procedure and options used. Provide bench records and all records of calibration, analyses, and calculations for standards, samples, blanks, any titration indicator blanks, duplicates, spikes, controls, etc. Include absorbances, peak heights, responses, concentrations, etc. for each measurement. Include digestion logs showing sample volumes and dilutions for all samples. Identify organic nitrogen compound used for matrix spikes. Records must be legible and sufficient to recalculate all concentrations and QA audit results. Provide photocopies of all instrument readouts (i.e. stripcharts, print-outs, etc). Report results as mg N/l. Identify the compound used for the matrix spike.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.
10. Other (use additional sheets or attach supplementary information, as needed): _____
11. Name of sampling/shipping contact: Marcia Kuehl
Phone: 414-458-8211 X 2253

I. DATA REQUIREMENTS

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (+% or Conc.)
TKN	0.1 mg N/l	Duplicate sample results must agree within 0.1 mg/l for concentrations <1 mg/l and within 10% for concentrations > or = to 1 mg/l
NOTE: These are minimum requirements. Report the actual detection limit used based on allowable methodology options.		

II. QC REQUIREMENTS Do not use designated field blanks for QA audits.

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
Control standards (Nicotinic Acid)	one per set	70 - 110% recovery
Matrix spike*	one per group of 10 or fewer samples	85 - 115% recovery
Lab duplicate	" "	+ (10% or 0.1 mg N/l)
Lab blank	" "	+ 0.1 mg N/l
Calibration verification Standard	" " and at the end of the set	90 - 110%
1 set of EPA QC nutrient reference samples conc. 3 and 4.	one per set	85 - 115%

*Matrix spike concentration will be greater than 30% of the sample concentration but will not exceed the highest calibration standard. Matrix spikes will be prepared from an organic nitrogen compound.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: Daily by overnight carrier
6. Number of days analysis and data required after laboratory receipt of samples: 30
7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
- 1) EPA Method 350.1 (Automated Phenate), or
- 2) EPA Method 350.3 (Potentiometric, Ion Selective Electrode).
- Samples will be stored at 4° C until analysis and validation of results. Sample aliquots will be preserved in the field with sulfuric acid (1 ml/l to pH < 2).
- The working concentration range of Method 350.1 Auto Analyzer should be 0.1 to 10 mg/l NH₃-N or lesser concentration.
8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
- Check sample pH (wide range pH paper). If pH > contact Jay Thakkar, CPMS, CRL for instructions. Dilute and rerun samples with peak heights or concentrations higher than the highest standard. The holding time is not to exceed 28 days from sample collection. All solutions should be made with ammonia-free water. For Method 350.3 calibrate the electrometer with standards in order of increasing concentration of ammonia. The pH of the solution after the addition of NaOH must be above 11. Use only the method(s) specified above. Standard curve for Method 350.1 must include at least 5 standards (one of which is zero concentration). Standard curve for Method 350.3 must include at least 4 standards between 0.1 and 10.0 mg/l NH₃-N. All standards, blanks, dilution water, and diluted samples must be acidified with 1 ml/l H₂SO₄.
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:
- The test procedure used will be clearly identified. Bench records tabulating the order of calibration standards, lab blanks, samples, lab control standards, spikes, duplicate, etc. with resulting peak heights, millivolts, or concentration readouts, will be provided along with copies of worksheets used to calculate ammonia results. If Method 350.3 is used, the standard curve should be provided. A photocopy of the instrument readout i.e. strip charts, printer tapes, etc. must be included. All records analyses and calculation must be legible and sufficient to recalculate all concentrations. Results are to be in mg/-N per liter. EPA OC reference samples, or any other reference sample or initial calibration verification will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.
10. Other (use additional sheets or attach supplementary information, as needed):
note if filtration needed, report which samples were filtered
11. Name of sampling/shipping contact: Marcia Kuehl
- Phone: 414-458-8711 x2253

NH₃-2-3-Analysis of ammonia
June 30, 1987**I. DATA REQUIREMENTS**

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (<u>±% or Conc.</u>)
<u>Ammonia</u>	<u>0.1 mg/l-N</u>	<u>Duplicate results must agree to within 10% for concentrations > 1mg/l or to within 0.1mg/l for concentrations < 1 mg/l</u>
<u>NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.</u>		<u>Results will be reported to the nearest 0.05 mg/l and to 2 significant figures for concentrations exceeding 1/mg/l-N.</u>

GENERAL STATEMENT**II. QC REQUIREMENTS - Do not use designated field blanks for QA Audits.****a) For Method 350.1**
Audits Required

<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
<u>at least 1 per group of 10 or fewer samples</u>	<u>85% - 115%</u>
<u>at least 1 per group of 10 or fewer samples</u>	<u>± 10% or 0.1 mg/l</u>
<u>at least 1 per group of 10 or fewer samples</u>	<u>≤ 0.1 mg/l</u>
<u>1 per group of 10 samples</u>	<u>90% - 110%</u>
<u>1 per sample set</u>	<u>85% - 115%</u>

b) For Method 350.3

<u>Lab Duplicate</u>	<u>at least 1 per group of 10 or fewer samples</u>	<u>10% or 0.1 mg/l</u>
<u>Lab Blank</u>	<u>at least 1 per group of 10 or fewer samples</u>	<u>≤ 0.1 mg/l</u>
<u>Calibration verification standard</u>	<u>1 per 10 samples and end of set</u>	<u>90% - 110%</u>
<u>1 set of EPA QC Nutrient reference samples. Conc. 1 & 2.</u>	<u>1 per sample set</u>	<u>85% - 115%</u>

*Matrix spike concentrations will be greater than 30% of sample concentrations, but spiked samples should not exceed working concentration range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

402-202-1
Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: daily by overnight courier
6. Number of days analysis and data required after laboratory receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1) EPA Method 353.1 (colorimetric, automated hydrazine reduction).

2) EPA Method 353.2 (colorimetric, automated cadmium reduction).

3) EPA Method 353.3 (colorimetric, manual cadmium reduction).

For all methods:

Samples will be stored at 4°C until analysis and validation of results. Samples will be preserved in the field with sulfuric acid (1 ml/l) to pH<2. The analytical working range shall not exceed 0.1 to 10.0 mg/l N.

For Methods 353.2 or 353.3: If more than one reduction column is used separate calibrations, QA audits, and records are required for each column. The column used must be identified for each analytical result.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Analyze the samples within 28 days after collection. Check the sample pH (wide range pH paper is acceptable). If the pH>2 contact CPMS, CRL for instructions. Use only the methods specified in item 7. Obtain approval of CPMS, CRL before using any other method.

For Methods 353.2 and 353.3: After checking the pH it is recommended that the laboratory check for residual chlorine (or oxidizing reagents) and sulfide using test strips such as starch iodide and lead acetate papers. Contact CPMS, CRL if these interferences are present; however, the laboratory must remove these interferences prior to analysis. The laboratory must also minimize interferences due to metals in order to prolong column life. (See Section 7.1.2 of method 353.3) It is suggested that the laboratory may dilute samples up to ten-fold prior to analysis (Section 7.4 of Method 353.3) provided that the final analytical working range does not exceed 0.1 to 10.0 mg/l N.

For all methods: Neutralize samples to pH 5-9 (or to phenolphthalein color end-point) prior to analysis. Dilute and reanalyze the neutralized samples if the concentrations exceed that of the highest standard. Use at least five calibration standards (including a zero standard). Prepare the lab blank using 1 ml of H₂SO₄/l. Neutralize and analyze it like a sample.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:

The test procedure used must be clearly identified. Bench records tabulating the order of calibration standards, lab control standards, lab blanks, samples, spikes, duplicates, etc., with resulting absorbances or concentration readouts will be provided. Worksheets used to calculate results will be included. Any sample treatment to remove interferences will be documented. The laboratory shall submit photocopies of the instrument readout (strip-charts, printer tapes, etc.) All records of analysis and calculations must be legible and sufficient to recalculate all concentrations. Results are to be reported as mg N/l.

EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

report which sample (if any) required prefiltration

11. Name of sampling/shipping contact: Marcia Kuenel

Phone: 414 458 8711 x2253

I. DATA REQUIREMENTS

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (+% or Conc.)
<u>Nitrate + Nitrite</u>	<u>0.10 mg/l as N</u>	<u>Duplicate results must be within 10% for concentrations >1mg/l or within 0.1 mg/l for concentrations < 1mg/l. Results will be reported to the nearest 0.1 mg/l for conc. less than 1.0 mg/l and to 2 significant figures for conc. exceeding 1 mg/l-N.</u>
<u>Note: These are minimum requirements. Report actual detection limits used based on allowable methodology options.</u>		

II. QC REQUIREMENTS - Do not use any designated field blanks for QA audits.

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
<u>Matrix Spike*</u>	<u>1 per group of 10 or fewer samples</u>	<u>85% - 115%</u>
<u>Lab Duplicate</u>	<u>1 per group of 10 or fewer samples</u>	<u>±(10% - or 0.10 mg/l)</u>
<u>Lab Blank (1ml/1 H₂SO₄)</u>	<u>2 per sample set</u>	<u><0.1 mg/l</u>
<u>Calibration verification standard</u>	<u>1 per group of 10 or fewer samples and at end of run</u>	<u>90% - 110%</u>
<u>Calibration blank</u>	<u>1 per group of 10 samples or less</u>	<u>< 0.1 mg/l</u>
<u>1 set of EPA Nutrient QC reference samples-conc. 1 and 2, or EPA F/NO₃ QC sample, WS series Conc. 1 and 2</u>	<u>1 per sample set</u>	<u>85% - 115%</u>

*Matrix spike concentrations will be 30% or larger, of sample concentrations, but spiked samples should not exceed working concentration range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: daily by overnight courier
6. Number of days analysis and data required after laboratory receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA Method 410.1 (Titrimetric, Mid-level) for COD > 50 mg/l.

EPA Method 410.2 (Titrimetric, Low-level) for COD ≤ 50 mg/l.

Use Section 7.1 of Method 410.3 if chloride concentration exceeds 2000 mg/l in a sample.

If titration blank is necessary for each different amount of mercuric sulfate used for inhibition of chloride interference, SAS Packing Lists will note the samples requiring assessment of chloride interferences. Measurement of chloride will be done using any method of "Standard Methods", 16th ed., or "EPA Methods for Chemical Analysis of Water and Wastes", 1983 ed., whenever possible chloride interference is noted.

Samples will be preserved with 1 ml of H_2SO_4 to pH less than 2 and kept at $4^\circ C$ until sample analysis and validation of results are completed. Holding time is not to exceed 28 days from date of sample collection.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Check sample pH (wide range pH paper). If $pH > 2$, contact CPMS, CRL for further instructions.
2. Use a) 50 ml sample aliquots for both methods, b) 0.250 N $K_2Cr_2O_7$ reagent and 0.25 N ferrous ammonium sulfate titrant for Method 410.1, and c) 0.0250 N $K_2Cr_2O_7$ reagent and 0.025 N ferrous ammonium sulfate titrant for Method 410.2.
3. Dilute and reanalyze (by Method 410.1) any samples with COD values > 800 mg/l or titrant volumes < 5.0 ml. Reanalyze samples (by Method 410.1) if initial sample values are > 50 mg/l COD by Method 410.2. Reanalyze samples (by Method 410.2) if initial sample values are < 50 mg/l COD by Method 410.1.
4. Any sample aliquots < 50 mls will be diluted to 50 mls so that the COD reaction mixture will be 50% H_2SO_4 / 50% water by volume.
5. Titration blanks will be determined, at least in duplicate each day of analysis and will not differ more than ± 0.1 ml titrant for Method 410.1 and ± 1.0 ml titrant for Method 410.2.
6. Separate sets of QA Audits will be performed for each method, if both methods are used.
7. Use potassium hydrogen phthalate as a matrix spike compound. Use 20 mg/l matrix spike concentration for Method 410.2.
8. Samples will be refluxed for at least 2 hours.
9. Homogenize sample aliquots, as necessary, to obtain sample aliquots of representative suspended solids.
10. Use only the method specified.

I. DATA REQUIREMENTS

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (+/- % or Conc.)
<u>COD (Method 410.1)</u>	<u>50 mg/l</u>	<u>Method 410.1: Differences in sample duplicates are to be < or = to 0.2 ml titrant or < 8 mg/l for concentrations < 80 mg/l and < 10% for COD concentrations exceeding 80 mg/l.</u>
<u>COD (Method 410.2)</u>	<u>5 mg/l</u>	
<u>NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.</u>		
		<u>Method 410.2: Differences in sample duplicate results are to be < 1.0 ml titrant or < 4 mg/l for concentrations less than 40 mg/l and are to be < 5 mg/l for concentrations between 40 50 mg/l.</u>

II. QC REQUIREMENTS

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
<u>Matrix spike (KHP)</u>	<u>at least 1 per group of 10 or fewer samples</u>	<u>85 - 115% Recovery (410.1)</u> <u>75 - 125% Recovery (410.2)</u>
<u>Method 410.1*</u>		
<u>Method 410.2(Use 20 mg/l spike)</u>		
<u>Lab duplicate</u>	<u>" "</u>	<u>Diff ≤ (8 mg/l or 10% (410.1)</u> <u>Diff ≤ (4 mg/l - 5 mg/l (410.2)</u>
<u>Titration blank (used for calculation of results)</u>	<u>at least 2 per sample set for each method used</u>	<u>Diff in titrant volumes shall not exceed 0.1 ml for 410.1 and 1.0 ml for 410.2</u>
<u>1 set of EPA QC Demand Reference samples - 2 concentration levels</u>	<u>1 per sample set for each method used</u>	<u>90 - 110% Recovery or < 8 mg/l error for 410.1 and < 5 mg/l error for 410.2 in aliquot - tested</u>

* - Matrix spike will be greater than 30% of the sample concentration, but spiked sample shall not exceed 800 mg/l for Method 410.1.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

COD-3⁽²⁾

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records, tabulating titrant standardization, titration volumes for titration or sample blanks (2 or more in number), samples, and QA Audits will be provided for each method used. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA Audit results.

Records of chloride analysis will be provided for any samples so specified on the RAS/SAS Traffic Report or SAS Packing List. Separate bench records will be provided for any COD determinations of high chloride samples (>2000 mg/l Cl) including weight of mercuric sulfate used, sample titration volume and titration blank volume for each sample type.

EPA QC Reference samples, or any other reference samples, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact:

Marisa Vuehl

Phone:

414-458-8711 x 2253

BROMIDE

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request

☐

Regional Transmittal

☐

Telephone Request

- A. EPA Region/Client: IV
- B. RSCC Representative: Jan Pels
- C. Telephone Number: (312) 353-2720
- D. Date of Request: _____
- E. Site Name: Hinco Dump, Elkhart Indiana

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

- General description of analytical service requested: Analysis of 77
low level groundwater samples for bromide.
Samples will be filtered in the field
through a 0.45 micron filter and are to be
analyzed within 48 hours of collection.
- Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):
77 whole aqueous samples, low level
(historical levels 0.7 - 7 mg/L)
- Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):
Superfund remedial investigation

4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: daily by overnight carrier
6. Number of days analysis and data required after laboratory receipt of samples: 30
7. Analytical protocol required (attach copy if other than a protocol currently used in this program): USGS method I-2057-85 "Anions, Ion-Exchange chromatographic, automated" analyze within 48 hrs of collection
8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): optimize ion chromatographic conditions to separate Bromide from nitrate + sulfate as only bromide is to be analyzed + reported using this SAS. Select separator + suppressor column for optimum bromide resolution.
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion. Report results as mg/L dissolved bromide. Bench records tabulating the order + preparation of calibration standards, calibration verification standards, calibration curves, lab control standards, lab blanks, lab duplicates, lab matrix spikes
10. Other (use additional sheets or attach supplementary information, as needed): _____
11. Name of sampling/shipping contact: Marcia Kuehl
Phone: () 444 45 1800 242 7601 x2253

✓ With associated chromatograms + concentrations reported by the integrator. A photocopy of the instrument readouts and the settings (conductivity ion chromatograph operating parameters) should be included. All records of analysis must be legible and sufficient to calculate all concentrations and final results.

12. Data Requirements

Parameter	Detection Limit	Precision Desired ($\pm\%$ or Concentration)
Bromide	0.10 mg/L	$\pm 10\%$ for bromide concentrations ≥ 0.50 mg/L
		otherwise ± 0.10 mg/L
		for concentrations < 0.5 mg/L

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Lab Blank	daily (at least 1 per 20 spls)	< 0.10 mg/L
Lab Control Sample	daily (at least 1 per 20 spls)	85-115% recovery
* matrix spike	one per 10 samples	85-115% recovery
lab duplicate	one per 10 samples	$\pm 10\%$ rpd
Calibration Verification Standard	daily (at least 1 per 20 samples)	90-110% recovery

14. Action Required if Limits are Exceeded

Take corrective action and reanalyze samples:
 Contact Sample Management Office (SMO)

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

* matrix spike concentration to be $\geq 30\%$ sample concentration.

Anions, ion-exchange chromatographic, automated

Parameters and Codes:

Anions, dissolved, I-2057-85 (see below)

Parameter	Code	Parameter	Code
Bromide (mg/L as Br)	71870	Nitrite (mg/L as N)	00613
Chloride (mg/L as Cl)	00940	Orthophosphate (mg/L as P)	00671
Fluoride (mg/L as F)	00950	Sulfate (mg/L as SO ₄)	00945
Nitrate (mg/L as N)	00618		

1. Application

1.1 This method may be used only for the determination of dissolved bromide, chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate in natural water. Table 11 shows approximate lower and upper concentration limits. Actual limits depend on many factors including the column age, which affects column resolution, the relative concentrations of closely eluting species, and the volume of the sample injected. Samples containing anion concentrations high enough to overload the column resins or interfere with closely eluting species need to be diluted or a sample loop smaller than the 200- μ L sample loop specified in this method needs to be used. Sample dilution or use of smaller volumes will change the detection limits for all anions.

1.2 Analyses must be performed on filtered and unacidified samples.

1.3 The ion chromatographic (IC) technology is so new that instruments and associated data-processing equipment and software available on the commercial market are not standardized and operating conditions vary enormously. Until operating conditions of various manufacturers' instruments become more comparable and the equivalency of methods using those instruments is established by extensive testing, the IC method approved for U.S. Geological Survey use will specify instrument and associated software brands. This does not imply endorsement of one product over another, but rather, acknowledges

Table 11.—Working ranges of anions by ion chromatography

Constituent	Minimum concentration ¹ (mg/L)	Maximum concentration (mg/L)
Fluoride	0.01	50
Chloride	.20	50
Nitrite-nitrogen	.02	70
Orthophosphate-phosphorus	.06	40
Bromide	.10	150
Nitrate-Nitrogen	.05	150
Sulfate	.20	100

¹With a larger sample loop (for example, 800 μ L), minimum concentration levels can be lowered.

that IC technology is rapidly changing and developing.

2. Summary of method

2.1 A sample is injected into an ion chromatograph and is pumped through three different ion-exchange columns into a specific-conductivity detector. The first two columns, a precolumn and separator column, are packed with low-capacity anion exchanger. Ions are separated based on their affinity for the exchange sites of the resin. The last column is a suppressor column that contains cation-exchange resin in the hydrogen form. The suppressor column reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample into their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell. Anions are identified based

on their retention times compared with known standards. Quantitation is accomplished by measuring the peak height or area and by comparing it with an analytical curve generated from known standards.

2.2 During analysis, the suppressor column will slowly be exhausted and, therefore, will need to be regenerated. Other suppressors, such as the hollow-fiber suppressor, which is continuously regenerated, may be used.

2.3 For additional information on ion chromatography, see Small and others (1975) and Fishman and Pyen (1979).

3. Interferences

3.1 Because bromide and nitrate elute very closely together, they potentially interfere with each other. Bromide-to-nitrate ratios should not exceed 1:10 or 10:1 if both ions are to be quantitated.

3.2 High levels of organic acids may be present in industrial and domestic wastes which may interfere with inorganic-anion analysis. Two common species, formate and acetate, elute between fluoride and chloride.

3.3 Water from the sample injection will cause a negative peak or dip in the chromatogram when it elutes, because its conductivity is less than that of the suppressed eluent. This dip usually occurs between F^{-1} and Cl^{-1} . Any peak of interest eluting near the water dip must be sufficiently resolved from the dip to be accurately quantitated. A method of eliminating the conductivity drop due to bicarbonate and carbonate is to introduce into the sample concentrations of bicarbonate and carbonate that closely approximate those of the eluent used for analysis. Adjustment of the sample background may be accomplished in two ways.

3.3.1 Dilute the sample with eluent if sample dilution is required prior to analysis.

3.3.2 A volume of 1.0 mL of a prepared eluent concentrate (a solution that is 100 times more concentrated than the eluent with respect to bicarbonate and carbonate ions) can be added per 100.0 mL of sample. CAUTION: Samples prepared in this manner have a pH of about 10 and will readily absorb carbon dioxide if left exposed to the atmosphere. The result will cause a positive-peak interference.

3.3.3 Standard solutions need to be prepared in the same manner as the samples. It is important

to prepare a blank using demineralized water at eluent strength in bicarbonate and carbonate to indicate any interferences that may have been introduced by the sample-preparation technique.

3.4 Samples containing high concentrations of chloride or other anions may prevent resolution of closely eluting peaks. For example, the peak for 0.1 mg of bromide per liter in the presence of greater than 1,000 mg of chloride per liter is swamped by the chloride peak. Bromide begins to elute before the chloride peak completely returns to the baseline.

3.5 Unexpected, late-eluting peaks are a potential source of interference. A peak eluting about two minutes after sulfate, believed to be oxalate, has been observed in some precipitation samples.

4. Apparatus

4.1 *Ion Chromatograph, Dionex Model 12; auto-sampler, Gilson; integrator (NOTE 1), Spectra Physics* using the following operating conditions:

Sample loop -----	200 μ L
Eluent flow rate -----	138 mL/h (30 percent of full capacity)
Sample pump flow rate	50 percent of full capacity

Specific conductance

meter settings ----- 10, 30, or 100 μ S

NOTE 1. A dual pen recorder (1 V and 100 mV) may replace an integrator. The recorder should be capable of full-scale response in two seconds or less. A typical chart speed is 0.5 cm/min.

4.1.1 *Precolumn*, 4 \times 50-mm, fast-run, anion-resin column (Dionex P/N 030831 or equivalent) placed before the separator column to protect the separator column from contamination by particulates or species strongly retained by the ion-exchange resin.

4.1.2 *Separator column*, 4 \times 250-mm, fast-run, anion-separator column packed with low-capacity, pellicular, anion-exchange resin (Dionex P/N 030830 or equivalent) that is styrene divinylbenzene-based. This is suitable for resolving fluoride, chloride, nitrite, orthophosphate, bromide, nitrate, and sulfate.

4.1.3 *Suppressor column*, 6 \times 250-mm, column-packed, with a high-capacity, column-exchange resin (Dowex 50W-X 16-H form resin or equivalent) that is capable of converting the

eluent and separated anions to their respective acid forms.

4.2 For additional information, refer to the different manufacturers' instruction manuals.

5. Reagents

5.1 *Eluent*, 0.003 M sodium bicarbonate-0.0024 M sodium carbonate: Dissolve 0.2520 g NaHCO_3 and 0.2544 g Na_2CO_3 in demineralized water and dilute to 1 L (NOTE 2).

NOTE 2: Eluent concentration may be varied slightly to obtain the same retention times for each anion when a new separator column is used. The NaHCO_3 is subject to thermal decomposition and must be weighed without prior drying.

5.2 *Suppressor regeneration solution*, 1N H_2SO_4 : Cautiously add 111 mL concentrated H_2SO_4 (sp gr 1.84) to approx 600 mL demineralized water. Cool and dilute to 4 L with demineralized water.

5.3 *Standard anion solutions*: Dry all salts for 1 h at 105°C unless otherwise specified. Store each standard solution in TFE-fluorocarbon bottles.

5.3.1 *Bromide standard solution*, 1.00 mL = 1.00 mg Br: Dissolve 1.2877 g NaBr in demineralized water and dilute to 1,000 mL.

5.3.2 *Chloride standard solution*, 1.00 mL = 1.00 mg Cl: Dissolve 1.6484 g NaCl in demineralized water and dilute to 1,000 mL.

5.3.3 *Fluoride standard solution*, 1.00 mL = 1.00 mg F: Dissolve 2.2101 g NaF in demineralized water and dilute to 1,000 mL.

5.3.4 *Nitrate-nitrogen standard solution*, 1.00 mL = 1.00 mg $\text{NO}_3\text{-N}$: Dissolve 6.0681 g NaNO_3 in demineralized water and dilute to 1,000 mL.

5.3.5 *Nitrite-nitrogen standard solution*, 1.00 mL = 1.00 mg $\text{NO}_2\text{-N}$: Dissolve 4.9259 g NaNO_2 in demineralized water and dilute to 1,000 mL.

5.3.6 *Phosphorus standard solution*, 1.00 mL = 1.00 mg P: Dissolve 4.3936 g anhydrous KH_2PO_4 in demineralized water and dilute to 1,000 mL.

5.3.7 *Sulfate standard solution*, 1.00 mL = 1.00 mg SO_4 : Dissolve 1.8140 g K_2SO_4 in demineralized water and dilute to 1,000 mL.

5.4 *Mixed stock solution*: Prepare 1,000 mL mixed stock solution by appropriate quantitative dilution of each standard solution (NOTES 3 and 4).

Anion	Concentration (mg/L)	Volume (mL)
F	5.00	5
Cl	50.0	50
$\text{NO}_3\text{-N}$	5.0	5
$\text{PO}_4\text{-P}$	5.0	55
Br	5.0	5
$\text{NO}_2\text{-N}$	50.0	50
SO_4	50.0	50

NOTE 3. If nitrite is omitted from the mixed stock solution, the solution is stable for at least 1 month when stored and refrigerated in a clean TFE-fluorocarbon bottle. If nitrite is included in the mixed-stock solution, the solution needs to be prepared fresh daily.

NOTE 4. The above is only an example of a mixed-stock solution. Other appropriate concentrations can be prepared.

5.5 *Mixed standard solutions*: Prepare at least three mixed standard solutions by appropriate dilution of the mixed stock solution. The solutions should bracket the concentration range of interest.

6. Procedure

6.1 Set up the ion chromatograph according to the operating parameters described in 4.1. Equilibrate the columns with eluent until a stable baseline is obtained. Allow approximately 30 min for equilibration.

6.2 Set the full-scale conductivity to 10, 30, or 100 μS as is appropriate for the expected sample-anion concentrations. The higher settings are required for higher sample-anion concentrations.

6.3 Level the integrator at 10 mV (a display of 1000 with no signal). Adjust the ion chromatograph's offset to approximately 11 mV (a display of 1100). This ensures that the ion chromatograph's signal will not fall below 10 mV during the course of the analyses. The baseline signal tends to drift in a negative direction over a long period of time. Each chromatogram can be started at a signal level of 10 mV using the integrator's automatic-zero control.

6.4 Enter an appropriate program into the main program controller of the ion chromatograph according to the manufacturer's instruction manual. The system is configured so that the ion chromatograph controls the autosampler and starts the integrator at the beginning of each sample injection (NOTE 5).

NOTE 5. For additional information on computerized data reduction, see Hedley and Fishman (1982).

6.5 Place the mixed standard solutions in the first positions of the sample tray followed by a standard reference material and then the samples. Place a standard reference material in every twentieth position of the remainder of the sample tray.

6.6 Create an information file in the integrator by pressing the DIALOG key. Through this information file, various integrator functions can be enabled or disabled during the recording of a chromatogram. The only necessary function is ER (end run). It terminates the chromatogram at the appropriate time as determined by the operator's setting of the ion chromatograph's controller, which actuates the sampler and causes the injection of a new sample.

6.7 Press the integrator's PT EVAL key before starting a series of analyses. The integrator will take about 50 s to store the baseline signal so that a peak can be distinguished from baseline noise. The baseline noise can be evaluated before each chromatogram, using the integrator's ET function.

6.8 Set the ion chromatograph's PGM/AUTO/MANUAL switch from MANUAL to AUTO and press Start/Step to begin the analyses.

7. Calculation

7.1 The integrator automatically computes the concentration of each anion in each sample by comparing its peak height or area to the analytical curve. Retention times for the seven anions are given in table 12.

8. Report

8.1 Report bromide (71870), chloride (00940), fluoride (00950), nitrate-nitrogen (00618), nitrite-nitrogen (00613), orthophosphate-phosphorus (00660), and sulfate (00945), dissolved, concentrations as follows: less than 1 mg/L, nearest 0.01 mg/L; 1 mg/L and above, two significant figures.

9. Precision

9.1 Analysis of a number of test samples 10 times each by one operator resulted in mean values, standard deviations, and percent relative standard deviations as shown in table 13.

Table 12.—Approximate retention times of anions by ion chromatography

Constituent	Time (min)
Fluoride	2.2
Chloride	3.3
Nitrate-nitrogen	4.0
Orthophosphate-phosphorus	4.9
Bromide	6.5
Nitrite-nitrogen	7.5
Sulfate	8.6

Table 13.—Precision for ion chromatographic determination of anions

Constituent	Mean (mg/L)	Standard deviation (mg/L)	Relative standard deviation (percent)
Bromide	0.295	0.020	6.8
Chloride	.72	.04	5.6
Do.	1.71	.06	3.5
Do.	2.72	.24	8.8
Do.	5.84	.19	3.2
Do.	9.90	.39	3.9
Do.	58.6	.7	1.2
Do.	119	1.2	1.0
Fluoride	.018	.004	22.2
Do.	.080	.010	12.5
Do.	.79	.02	2.5
Do.	.92	.01	1.1
Do.	2.02	.15	7.4
Nitrate-nitrogen	.12	.01	8.3
Do.	.42	.051	1.9
Do.	.70	.081	1.4
Do.	1.27	.05	3.9
Do.	5.26	.14	2.7
Nitrite-nitrogen	.03	.01	33.3
Orthophosphate-phosphorus	.273	.010	3.7
Sulfate	1.68	.05	3.0
Do.	3.88	.10	2.6
Do.	15.1	.80	5.3
Do.	62.1	.9	1.4
Do.	100	1.4	1.4
Do.	146	3	2.0

References

- Fishman, M. J., and Pyen, G. S., 1979, Determination of selected anions in water by ion chromatography: U.S. Geological Survey Water-Resources Investigations, 79-101, 30 p.
- Hedley, A. G., and Fishman, M. J., 1982, Automation of an ion chromatograph for precipitation analysis with computerized data reduction: U.S. Geological Survey Water-Resources Investigations, 81-78, 33 p.
- Small, H., Stevens, T. S., Bauman, W. C., 1975, Novel ion exchange chromatographic method using conductimetric detection: *Analytical Chemistry*, v. 47, p. 1801-9.

res.wells org.SAS

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES Client Request



Regional Transmittal



Telephone Request

- A. EPA Region/Client: V/
- B. RSCC Representative: Jan Pels
- C. Telephone Number: (312) 353-2720
- D. Date of Request: _____
- E. Site Name: Hinco Dump, Elkhart Indiana

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for our request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

- General description of analytical service requested: Analysis of drinking water/
residential wells for volatiles, semi-volatiles, and pesticides/PCBs with low
quantitation limits.
- Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):
17 low level residential well waters -VOA (1 trip blank)
16 low level residential well waters - BNA, PCB/Pest
- Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):
Superfund Remedial Investigation

4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: daily by overnight courier
6. Number of days analysis and data required after laboratory receipt of samples:
7 days for analysis. Final report and data due within 15 days.
7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
CLP SOW for Organic Analysis (Multi-Media, Multi-concentration) ~~2/88~~ 2/88.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
1. Modifications to the CLP Organic SOW ^{2/88} ~~2/87~~ in Attachment I.
2. Required low level quantitation limits in Attachment II.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.
^{2/88}
As per CLP Organics SOW ~~2/87~~ with modifications as outlined in Attachment I.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Marcia Kuehl
Phone: 414-458-8711 x2253

I. DATA REQUIREMENTS

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired (+%, or Conc.)</u>
<u>See attachment II</u>	<u>See attachment II</u>	<u>± 20%</u>
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

II. QC REQUIREMENTS

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
<u>As per CLP Organic SOW ^{2/88} 8/88</u>	<u>As per CLP Organic SOW ^{2/88} 8/88</u>	<u>Exceptions to CLP Organic SOW ^{2/88} 8/88: See attachment I.</u>
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact SMO

ATTACHMENT 1 (page 1 of 2)
MODIFICATIONS TO THE CLP ORGANICS SOW 8/87
FOR THE REGION V RESIDENTIAL WELL SAS

1. Special Technical Instructions.

Volatile Analysis

- a) Sample volume is increased from 5 ml to 20 or 25 ml to achieve lower detection limits. Lab must use the same volume of sample that is used for the MDL study.
- b) Acrolein and acrylonitrile are additional target compounds. These compounds will be added to both initial and continuing calibration mixes. Acrolein and acrylonitrile will use bromochloromethane as internal standard. The primary and secondary quantitation ions to be used for these compounds are:

<u>Compound</u>	<u>Primary Ion</u>	<u>Secondary Ion(s)</u>
Acrolein	56	55
Acrylonitrile	53	52
- c) Initial calibration: Five point initial calibration - 5, 10, 20, 40 and 60 ug/L for all compounds except for acrolein and acrylonitrile, which should be analyzed at 25, 50, 75, 100 and 125 ug/L.
- d) Continuing calibration: Performed at 20 ug/L for all compounds except acrolein and acrylonitrile, which should be analyzed at 50 ug/L.
- e) Surrogate standards: SOW standards spiked at 20 ppb.
- f) Matrix spike/matrix spike duplicate: Matrix spike compounds at 20 ppb.

Semivolatiles Analysis

- a) Extraction/blowdown: Extract the entire one liter bottle, rinsing the cap & bottle and add to sample. Final blowdown volume may be decreased to 0.5 ml to achieve required detection limits.
- b) Initial Calibration: Five points at 5, 10, 20, 50, and 100 total nanograms except for benzoic acid, 2,4-dinitrophenol, 2,4,5-trichlorophenol, all three nitroaniline isomers, 4-nitrophenol, 4,6-dinitro-2-methylphenol and pentachlorophenol which should be analyzed at 20, 50, 80, 100 and 120 total nanograms. The laboratory should notify the Region if this calibration range is not possible.
- c) Continuing calibration: 20 total nanograms except for benzoic acid, 2,4-dinitrophenol, 2,4,5-trichlorophenol, all three nitroaniline isomers, 4-nitrophenol, 4,6-dinitro-2-methylphenol and pentachlorophenol which should be analyzed at 50 total nanograms.
- d) Surrogate standards: SOW standards spiked as 20ppb for base-neutral standards and 40ppb for the acid standards.
- e) Matrix spike/matrix spike duplicate: Matrix spike compounds as per the SOW spiked at 20ppb for base-

neutrals and 40ppb for the acids.

ATTACHMENT 1

(page 2 of 2)

Pesticides/PCBs Analysis

- a) Extraction/blowdown: Extract the entire one liter bottle; rinse cap & bottle and add to sample. Final blowdown volume may be decreased up to 0.5mL to achieve required detection limits.
 - b) Surrogate standard: Spiked at 0.2 ppb.
 - c) Matrix spike/matrix spike duplicate: SOW Matrix spike compounds spiked at 1/5 the SOW concentration.
2. Analytical Results Required.
- a. Quantitation Limits: Organic Analysis Data Sheets, Form 1 will reflect method detection limits experimentally determined and verified previously by the contractor.
 - b. Additional volatile parameters: Results will include all data for acrolein/acrylonitrile analogous to the other volatile target compounds in the SOW. Form 1s may be appended to include these parameters.
 - c. Dilutions: The contractor will request permission of the Region to dilute any sample exceeding the initial calibration range for any parameter. Diluted and undiluted sample data will be included in the results as per the SOW.
3. QC Requirements:
- a. Initial Calibration (Volatiles/Semivolatiles):
SPCC criteria apply.
The %RSD for the RFs for all compounds must be $\leq 35\%$.
The RFs for all other (non SPCC) compounds must be ≥ 0.05 .
 - b. Continuing Calibration:
SPCC criteria apply.
The %D for the RFs for all compounds must be $\leq 25\%$.
The RFs for all other (non SPCC) compounds must be ≥ 0.05 .
 - c. Matrix Spike/ Matrix Spike Duplicates:
SOW criteria apply for %R and %RPDs.
 - d. Surrogates:
Sow criteria apply for %R and corrective action.
 - e. Blanks:
Sow criteria apply, however, the calculated MDLs must be used instead of RAS CRQLs.

ATTACHMENT II

1 of 4

(ALL UNITS ARE MICROGRAMS/LITER)

ANALYTE	CAS #	QUANTITATION LIMIT
BENZENE	71-43-2	1.5
BROMODICHLOROMETHANE	75-27-4	1.5
BROMOFORM	75-25-2	1.5
BROMOMETHANE	74-83-9	1.5
CARBON TETRACHLORIDE	56-23-5	1.5
CHLOROBENZENE	106-30-7	1.5
CHLOROETHANE	75-00-3	1.5
CHLOROFORM	67-66-3	1.5
CHLOROMETHANE	74-87-3	1.5
DIBROMOCHLOROMETHANE	124-48-1	1.5
1,1-DICHLOROETHANE	75-34-3	1.5
1,2-DICHLOROETHANE	107-06-2	1.5
1,1-DICHLOROETHENE	75-35-4	1.5
total 1,2-DICHLOROETHENE		1.5
1,2-DICHLOROPROPANE	78-87-5	1.5
cis-1,3-DICHLOROPROPENE	10061-01-5	2
trans-1,3-DICHLOROPROPENE	10061-02-5	1
ETHYL BENZENE	100-41-4	1.5
METHYLENE CHLORIDE (*)	75-09-2	1
1,1,1,2-TETRACHLOROETHANE	75-34-5	1.5
TETRACHLOROETHENE	127-18-4	1.5
TOLUENE (*)	108-88-3	1.5
1,1,1-TRICHLOROETHANE	71-55-5	1.5
1,1,2-TRICHLOROETHANE	79-00-5	1.5
TRICHLOROETHENE	75-01-5	1.5
VINYL CHLORIDE	75-01-4	1.5
ACROLEIN	107-02-8	2.5
ACETONE (*)	67-64-1	5
ACRYLONITRILE	107-13-1	2.5
CARBON DISULFIDE *	75-15-0	3
2-BUTANONE (*)	78-93-3	5
VINYL ACETATE	108-05-4	5
4-METHYL-2-PENTANONE	108-10-1	1.5
2-HEXANONE	519-78-6	5
STYRENE	100-42-5	1
m-XYLENE **	106-38-3	} 1.5
o-XYLENE **	95-47-6	
p-XYLENE **	106-42-3	

* COMMON LABORATORY SOLVENT
BLANK LIMIT IS 5x METHOD DETECTION LIMIT

** THE ^{m-xylylene} o-XYLENE AND p-XYLENE ARE REPORTED AS A TOTAL OF THE ^{three.} ~~one~~

ATTACHMENT II

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(ALL UNITS ARE MICROGRAMS/LITER)

ANALYTE	CAS #	QUANTITATION LIMIT
BIS(2-CHLOROETHYL)ETHER	111-44-4	1.5
PHENOL	108-95-2	2
2-CHLOROPHENOL	95-57-8	2
1,3-DICHLOROBENZENE	541-73-1	2
1,4-DICHLOROBENZENE	106-46-7	2
1,2-DICHLOROBENZENE	95-50-1	2.5
BENZYL ALCOHOL	100-51-5	2
BIS(2-CHLOROETHOXY) ETHER	333-33-3	2.5
2-METHYLENEOL	95-58-7	1
HEXACHLOROCYCLOHEPTANE	67-72-1	2
N-NITROSODIPROPYLAMINE	621-54-7	1.5
NITROBENZENE	98-95-3	2.5
4-METHYLPHENOL	106-44-5	1
ISOPHORONE	78-59-1	2.5
2-NITROPHENOL	88-72-5	2
2,4-DIMETHYLPHENOL	105-57-5	2
BIS(2-CHLOROETHOXY)METHANE	111-91-1	2.5
2,4-DICHLOROPHENOL	120-83-2	2
1,2,4-TRICHLOROBENZENE	120-82-1	2
NAPHTHALENE	91-20-3	2
4-CHLOROANILINE	106-47-8	2
HEXACHLOROBUTADIENE	87-68-3	2.5
BENZOIC ACID	65-25-0	20
2-METHYLNAPHTHALENE	91-57-5	2
4-CHLORO-2-METHYLPHENOL	59-50-7	1.5
HEXACHLOROCYCLOPENTADIENE	77-47-4	2
2,4,6-TRICHLOROPHENOL	88-06-2	1.5
2,4,5-TRICHLOROPHENOL	95-93-4	1.5
2-CHLORONAPHTHALENE	91-58-7	1.5
ACENAPHTHYLENE	208-96-8	1.5
DIMETHYL PHTHALATE	131-11-3	1.5
2,6-DINITROTOLUENE	606-20-2	1
ACENAPHTHENE	83-32-9	1.5
3-NITROANILINE	99-09-2	2.5
DIBENZOFURAN	132-64-9	1
2,4-DINITROPHENOL	51-29-5	(15)
2,4-DINITROTOLUENE	121-14-2	1

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(CONTINUED) (ALL UNITS ARE MICROGRAMS/LITER)

ANALYTE	CAS *	QUANTITATION LIMIT
FLUORENE	86-73-7	1
4-NITROPHENOL	100-02-7	1.5
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	1
DIETHYL PHTHALATE	84-56-2	1
4,6-DINITRO-2-METHYLPHENOL	534-52-1	(15)
N-NITROSO-2-ETHYLAMINE *	85-30-5	
DIPHENYLAMINE *	122-39-4	1.5
4-NITROANILINE	100-01-5	3
4-BROMOPHENYL PHENYL ETHER	101-55-3	1.5
HEXACHLOROBENZENE	118-74-1	1.5
PENTACHLOROPHENOL	87-86-5	2
PHENANTHRENE	85-01-8	1
ANTHRACENE	120-12-7	2.5
DI-n-BUTYL PHTHALATE	84-74-2	2
FLUORANTHENE	206-44-0	1.5
PYRENE	129-00-0	1.5
BUTYL BENZYL PHTHALATE	85-58-7	3.5
CHRYSENE **	218-01-9	
BENZO(a)ANTHRACENE **	56-55-3	1.5
BIS(2-ETHYL-HEXYL) PHTHALATE	117-81-7	1
DI-n-OCTYL PHTHALATE	117-84-0	1.5
BENZO(b)FLUORANTHENE ***	205-39-2	
BENZO(k)FLUORANTHENE ***	207-06-9	1.5
BENZO(a)PYRENE	50-32-8	2
INDENO(1,2,3-cd)PYRENE	193-39-5	3.5
DIBENZO(a,h)ANTHRACENE	53-70-3	2.5
BENZO(g,h,i)PERYLENE	191-24-2	4
2-NITROANILINE *	88-74-4	1

* THESE TWO PARAMETERS ARE REPORTED AS A TOTAL

** THESE TWO PARAMETERS ARE REPORTED AS A TOTAL

*** THESE TWO PARAMETERS ARE REPORTED AS A TOTAL

VALUES IN PARENTHESES ARE ESTIMATES. ACTUAL VALUES ARE BEING DETERMINED AT THIS TIME.

Attachment II

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Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

Pesticides/PCBs	CAS Number	Quantitation Limits**
		Water ug/L
alpha-BHC	319-84-6	0.010
beta-BHC	319-85-7	0.005
delta-BHC	319-86-8	0.005
gamma-BHC (Lindane)	58-89-9	0.005
Heptachlor	76-44-8	0.030
Aldrin	309-00-2	0.005
Heptachlor epoxide	1024-57-3	0.005
Endosulfan I	959-98-8	0.010
Dieldrin	60-57-1	0.010
4,4'-DDE	72-55-9	0.005
Endrin	72-20-8	0.010
Endosulfan II	33213-65-9	0.010
4,4'-DDD	72-54-8	0.020
Endosulfan sulfate	1031-07-8	0.10
4,4'-DDT	50-29-3	0.020
Methoxychlor	72-43-5	0.020
Endrin ketone	53494-70-5	0.030
alpha-Chlordane	5103-71-9	0.020
gamma-Chlordane	5103-74-2	0.020
Toxaphene	8001-35-2	0.25
Aroclor-1016	12674-11-2	0.10
Aroclor-1221	11104-28-2	0.10
Aroclor-1232	11141-16-5	0.10
Aroclor-1242	53469-21-9	0.10
Aroclor-1248	12672-29-6	0.10
Aroclor-1254	11097-69-1	0.10
Aroclor-1260	11096-82-5	0.10

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res. wells ino SAS

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Drinking Water (Inorganics)

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

Approved For Scheduling

SPECIAL ANALYTICAL SERVICES
Client Request

APR 25 1988

Approved for Scheduling

☒

Regional Transmittal



Telephone Request

- A. EPA Region/Client: V
- B. RSCC Representative: Jon Pels
- C. Telephone Number: (312) 353-2720
- D. Date of Request: _____
- E. Site Name: Hino Dump, Elkhart, Indiana

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of Drinking Water
and/or residential well water for metals and cyanide using detection limits lower than SO4

5502504787 (See Attachment II) Six elements are to be determined by GFAA using the method of standard additions. GFAA analysis of samples free of particulates may be conducted on the undigested sample.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

16 low level residential well waters

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund Remedial Investigation

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4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: daily by overnight courier
6. Number of days analysis and data required after laboratory receipt of samples:
7 days for analysis, Final Report + data due in 15 days.
7. Analytical protocol required (attach copy if other than a protocol currently used in this program): Inorganic analysis as per SOW 785, IFB WA-85-J838, with the exceptions listed in Attachments II & III. ICP emission spectroscopy, mercury, and cyanide analyses follow the SOW mentioned above for sample preparation and analysis protocol with the instrument detection limits and matrix spike levels given in Attachment II and the QC audits as described in Attachment III. GFAA analyses may be run undigested if the samples are free of particulates. If particulates are present the samples are to be digested as per SOW mentioned above. The ICP digest is to be used for Sb analyses, if digestion is required. A detailed set of instructions for conducting the GFAA analyses are included in Attachment III. Special instrument detection limits and matrix spike levels are listed on Attachment II.
8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): 1) Check the pH of each sample (wide range pH paper is acceptable). If the pH values are outside of the specified limits of SOW785, contact Region V for instructions.
2) Instrument Detection Limits (IDL) of Attachment II are to be met prior to any sample analysis. 3) Spike Ca, Mg, Na and K and all other parameters as per Attachment II. The Spikes for these four analytes shall be to a separate aliquot unless documentation is provided that no contamination results for the other analytes.
- The GFAA protocol is specified in Attachment III. The frequency and limits of certain audits are changed from that given in SOW 785 for all analyses as per Attachment III.
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.
- of CURRENT SOW
All of the deliverables included in SOW785 are required. Also, provide current quarterly XI, XII, XIII for each case. Submit Form VIII separate for each separate parameter analyzed by MSA. Form VIII must be modified to include the slope of each addition as well as the correlation coefficient. Use footnotes on Form I for reporting results, except use IDL of Attachment II for detection limit. MAKE CHANGES ON FORM I, XI, XII + O REFLECT SAS CONTRACT LIMITS AND IDL WHERE APPROPRIATE.
10. Other (use additional sheets or attach supplementary information, as needed): _____
11. Name of sampling/shipping contact: Marcia Kuehl
Phone: 414-458-8711 X2253

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance please call the Sample Management Office.

I. DATA REQUIREMENTS

<u>Parameter:</u>	<u>Detection Limit</u>	<u>Precision Desired</u> (+/- % or Conc.)
<u>ICP Metals</u>	<u>See Attachment II</u>	<u>10% RPD or Duplicate</u>
<u>Furnace Metals</u>	<u>" "</u>	<u>Differences < SAS IDL</u>
		<u>of - Attachment II</u>
<u>Mercury, Cyanide</u>	<u>See Attachment II as per</u>	<u>SOW 785</u>

II. QC REQUIREMENTS

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits* (% or Conc.)</u>
<u>For ICP -AES, Hg, and CN</u>	<u>See 9.A of Attachment III</u>	
<u>GFAA (undigested) As, Cd, Pb, Sb, Se, Ti</u>	<u>See 9.B of Attachment III</u>	
<u>GFAA (digested) As, Cd, Pb, Sb, Se, Ti</u>	<u>See 9.C of Attachment III</u>	

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and repeat analysis. contact SMO

ATTACHMENT II

Instrument Detection Limit and Spiking Level for Drinking Water

Compound	Required Instrument Detection Limit ¹ ug/L			Required Matrix Spike Concentrations ug/L		
	GFAA	ICP	Other	GFAA	ICP	Other
Metal:						
1. Aluminum		100			2000	
2. Antimony ²	5			20	500	
3. Arsenic	5			20		
4. Barium		50			2000	
5. Beryllium		5			50	
6. Cadmium ²	0.5			2	50	
7. Calcium ³		1000			50,000	
8. Chromium		10			200	
9. Cobalt		10			500	
10. Copper		10			250	
11. Iron		100			1000	
12. Lead ²	2			20	500	
13. Magnesium ³		1000			25,000	
14. Manganese		10			200	
15. Mercury			0.2			1.0
16. Nickel		20			400	
17. Potassium ³		2000			20,000	
18. Selenium	2			10		
19. Silver		5			50	
20. Sodium ³		1000			50,000	
21. Thallium	2			20		
22. Vanadium		10			500	
23. Zinc		20			200	
24. Cyanide			10			100

¹ Instrument Detection Limits (IDL) must be met before any samples are analyzed. The Lab may submit their quarterly Form XI with each case if all IDLs meet the detection limits. *IF Detection Limits cannot be met by USING ICP, USE OF GFAA REQUIRED.*

² ICP analysis results may only be reported for Sb, Cd and Pb, if the concentration is ≥ 10 times the IDL of instrument used. If ICP results are reported, all ICP audits are required including matrix spike.

³ Report Ca, Mg, Na and K on separate Form V for Matrix Spike if a separate aliquot is used for this spike.

ATTACHMENT III

Special instructions for GFAA and QC requirements for all analyses.

1. Sample aliquots are preserved in the field as follows:
 - a) One liter preserved with 5ml/l of 50% HNO_3 to $\text{pH} < 2$ for all metals (excluding Hg).
 - b) One liter sample preserved with 0.5% HNO_3 V/V and 0.05% $\text{K}_2\text{Cr}_2\text{O}_7$ W/V for Mercury.
 - c) One liter of sample preserved with 5ml/l of 6N NaOH to $\text{pH} \geq 12$ for cyanide determination.
2. Analysis of the six metals (specified in Attachment II) by graphite furnace atomic absorption (GFAA) must use the method of standard additions for quantitation.
3. All of the samples for GFAA metals can be analyzed without digestion if the samples are clean and without any particulates. In this case, a calibration blank, duplicate, ICVS, and CCVS shall be analyzed without digestion. *IF CCV is Out, Return Previous TO Samples and CV*
4. If any of the samples contain particulate or significant suspended solids, sample aliquots, preparation blank, duplicate, matrix spikes and lab control samples are to be digested per page D-2 of SOW785. The samples digested for ICP analysis are to be used for antimony determination.
5. No identified field blank may be used as a laboratory duplicate or matrix spike sample

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ATTACHMENT III

- 6.1 Zeeman, Smith/Hieftje background correction or equivalent (not D₂) is required for Arsenic, Selenium and Antimony or any element with structured background interferences.
- 6.2 The matrix modifiers of SOW785 are mandatory for As & Se.
- 6.3 L'vov platform is allowed.
- 6.4 Any matrix modifiers for Sb, Cd, Pb and Tl must be approved by the Region V Central Regional Laboratory's Contract Project Management Section prior to use and documented with the raw data.
- 6.5 Each sample or QC audit is to be determined by the MSA using the sample or QC audit and then three consecutive spikes.
- 6.6 Each calibration blank and QC audit solution must contain the same nitric acid concentration as the sample (or diluted samples). All solutions analyzed must have their matrix concentrations fully documented in the raw data.
- 6.7 Each analytical determination must have the resulting absorbance clearly recorded and documented in the order of determined.
- 8 The data for each MSA determination must show; slope (signal/conc.), intercept and correlation coefficient (r). The results must be reported on Form VIII for all samples and QC audits in order of analysis. Form VIII must be modified to include the above mentioned slope.
- 6.9 Samples and QC audits will be tested in the following order for the method of standard addition quantitation.
- a) calibration blank and + 3 spikes
 - b) ICVS (provided by EMSL-LV) +3 spikes
 - c) 5 samples, each with 3 spikes
 - d) calibration blank + 3 spikes
 - e) CCVS + 3 spikes
 - f) succeeding sets of 5 samples, cal. blank, and CCVS.
7. Report the correlation coefficient for all MSA analyses. $r \geq 0.995$ is required for all sample and audit analyses. A correlation coefficient $(r) \geq 0.998$ is recommended for the calibration blank or problems will occur with the sample analysis. If $r < 0.995$ or the slope is $< 35\%$ of the initial calibration blank, reanalyze the sample once. If the standard addition again fails these criteria, *Dilute sample 1:1 or more and reanalyze.* If the standard addition again fails, flag the data with a "+".
8. Care must be taken to avoid exceeding the linear range for all GFAA analyses. This problem is especially severe with Cd and Pb. Dilution of the samples may be necessary to avoid this problem. //
- 1 IF Sample Concentration Higher than the Highest Spike Added
Dilute Sample 1:1 and Reanalyzed
- 10 For MSA, use 10, 20, and 30 µg/L calibration standards, and for Cd, use 1, 2 and 3 µg/L calibration standards for 3 spikes Addition.

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9. A ICP Metals, Mercury and Cyanide

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits</u>
ICVS, CCVS, ICP serial dilution, ICP ICS, Distilled CN standard	as per SOW 785	as per SOW 785
Calibration Blank	Beginning of Run and 1 in 10 thereafter	≤ IDL
Preparation Blank	1 in 10 samples	< SAS IDL of Attachment II
Duplicate	1 in 10 samples	10% RPD or Difference is ≤ SAS IDL, 15% For Hg & CN
Matrix Spike (ICP)	1 in 10 samples	85 - 115% Recovery
Matrix Spike (ICP-Ca, Mg, Na, K)*	1 in 10 samples	85 - 115% Recovery
Matrix spike (Hg & CN)	1 in 10 samples	80 - 120%
Digested Lab Control Sample	1 per sample set	85 - 115%
*May be combined with other spike (cf item 8 of SAS)		

9. B G.F.A.A. Undigested Samples

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits</u>
1) Duplicate	1 in 10 samples	Difference of < SAS IDL of Attachment II or ≤ 10% RPD

2) Calibration Blank	Initially and after every 5 samples	≤ IDL
----------------------	-------------------------------------	-------

3) ICVS and CCVS	Initially ICVS, and CCVS after every 5 samples	90% - 110%
------------------	------------------------------------------------	------------

9. C GFAA Digested Samples

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits</u>
1) Calibration Blank	Initially and after every 5 samples	≤ IDL
2) Preparation Blank (Digested)	1 in 10 samples	< SAS IDL of Attachment II
3) Duplicates (Digested)	1 in 10 samples	Difference of < SAS IDL or 10% RPD
4) Matrix Spike (Digested)	1 in 10 samples	85 - 115% Recovery
5) Lab Control Sample (Digested)	1 per set of samples	85 - 115% Recovery
6) ICVS, CCVS	Initially ICVS, and CCVS after every 5 samples	90 - 110% Recovery

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request

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Regional Transmittal

☐

Telephone Request

- A. EPA Region/Client: V
- B. RSCC Representative: Jan Pels
- C. Telephone Number: (312) 353-2720
- D. Date of Request: _____
- E. Site Name: Himco Dump, Elkhart, Indiana

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of 17 pairs of Tenax & Tenax/charcoal sorbent tubes for the volatile organic compounds listed in Table 1 and up to 10 TICs. Each pair is to be analyzed in series. Two pairs are to be analyzed within 24 hours of receipt.
2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):
19 low level landfill gas samples collected on Tenax & Tenax/charcoal sorbent tube pairs. Two pairs are to be analyzed within 24 hours of receipt.
3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.): Superfund Remedial Investigation.

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: Daily by overnight carrier.

6. Number of days analysis and data required after laboratory receipt of samples:
17 samples: Analyze within 7 days of receipt, report results within 30 days
of receipt of samples. 2 samples: Analyze and report verbal results within
24 hours of receipt, hard copy data package due 30 days after receipt.
7. Analytical protocol required (attach copy if other than a protocol currently used in
this program): Protocol for Analysis of Sorbent Cartridges from
Volatile Organic Sampling Train EPA SW-846 Method 5040 Third Edition
(attached) and see also Attachment II and III.

8. Special technical instructions (if outside protocol requirements, specify compound
names, CAS numbers, detection limits, etc.): See attached (Attachments I,
II, and III).

9. Analytical results required (if known, specify format for data sheets, QA/QC
reports, Chain-of-Custody documentation, etc.) If not completed, format of results
will be left to program discretion. See Attachment IV. See Attachment III
for units to be used.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Marcia Kuehl
Phone: (414) 458-8711

8. Special Technical Instructions

Laboratory will supply:

27 pairs of Tenax and Tenax/charcoal tubes:

- 19 for field samples
- 2 for field duplicates
- 2 for tube "bottle" blanks
- 2 for upwind sample locations
- 1 for matrix spike
- 1 for matrix spike duplicate
- 3 extra for pump calibration and breakage

Tubes will comply with the I/I type listed in Attachment I. The laboratory shall specify the inner diameter of the cartridge. The cost of the Tenax, charcoal, glass tubes, and cleaning/conditioning and certifying the cartridges are the responsibility of the CLP SAS lab. The tubes will be prepared and shipped to the site no more than 2 weeks prior to sampling. In addition to cartridge preparation, the SAS lab shall provide all necessary fittings and adapters to allow field sampling using an air sampling pump with a 1/4-inch sampling port. All pre-cleaned and exposed cartridges will be stored at -20°C intact with end caps and placed in culture tubes with cleaned charcoal. Blank concentrations should not be subtracted from sample results. Each cartridge pair should be analyzed in series. Verify the detection limits through a cartridge spiked with all target compounds at the concentration of the detection limit as indicated in Attachment II.

Laboratory must uniquely label each tube and send a completed chain-of-custody form with the cartridges to the field, and use custody seals on all shipping containers.

12. Data Requirements

<u>Parameter</u>	<u>Detection Limit</u>	<u>Precision Desired (±% or Concentration)</u>
CLP TCL VOAs	5 ng/tube	+25%
(Table 1 Attachment I)		

13. QC Requirements

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits (Percent or Concentration)</u>
Method Blank*	daily, at start and every 12 hour shift	5 ng each TCL
Continuing Calibration Check	daily, at start and every 12 hour shift	+25%
MS, MSD	1 per group of 10	50-150% recovery 25% rpd
Surrogate Spike	all samples, STDS blanks	50-150% recovery document with data package +25%

14. Detection Verification daily
Action Required if Limits are Exceeded

Contact SMO.

*Method Blank consists of an unexposed Tenax cartridge and an unexposed Tenax/charcoal cartridge.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

ATTACHMENT I

Sorbent Tube Specifications

Source: Method 0030, SW-846, Third Edition

2.1.4 Sorbent cartridges:

2.1.4.1 The sorbent cartridges used for the VOST may be used in either of two configurations: the inside-outside (I/O) configuration in which the cartridge is held within an outer glass tube and in a metal carrier, and the inside-inside (I/I) configuration in which only a single glass tube is used, with or without a metal carrier. In either case, the sorbent packing will be the same.

2.1.4.1.1 The first of a pair of sorbent cartridges shall be packed with approximately 1.6 g Tenax GC resin and the second cartridge of a pair shall be packed with Tenax GC and petroleum-based charcoal (3:1 by volume; approximately 1 g of each).

2.1.4.1.2 The second sorbent cartridge shall be packed so that the sample gas stream passes through the Tenax layer first and then through the charcoal layer.

2.1.4.2 The sorbent cartridges shall be glass tubes with approximate dimensions of 10 cm by 1.6 cm I.D. The two acceptable designs (I/O, I/I) for the sorbent cartridge are described in further detail below.

2.1.4.2.1 Inside/Inside sorbent cartridge: A diagram of an I/I sorbent cartridge is shown in Figure 3. This cartridge is a single glass tube (10 cm by 1.6 cm I.D.) which has the ends reduced in size to accommodate a 1/4- or 3/8-in. Swagelok or Cajon gas fitting. The resin is held in place by glass wool at each end of the resin layer. The amounts of each type of sorbent material used in the I/I design are the same as for the I/O design. Threaded end caps are placed on the sorbent cartridge after packing with sorbent to protect the sorbent from contamination during storage and transport.

2.1.4.2.2 Inside/Outside type sorbent cartridge: A diagram of an I/O sorbent cartridge is shown in Figure 4. In this design the sorbent materials are held in the glass tube with a fine mesh stainless steel screen and a C-clip. The glass tube is then placed within a larger diameter glass tube and held in place using Viton O-rings. The purpose of the outer glass tube is to protect the exterior of the resin-containing tube from contamination. The two glass tubes are held in a stainless steel cartridge holder, where the ends of the glass tubes are held in place by Viton O-rings placed in machine grooves in each metal end piece. The three cylindrical rods are secured in one of the metal end pieces and fastened to the other end piece using knurled nuts, thus sealing the glass tubes into the cartridge holder. The end pieces are fitted with a threaded nut onto which a threaded end cap is fitted with a Viton O-ring seal, to protect the resin from contamination during transport and storage.

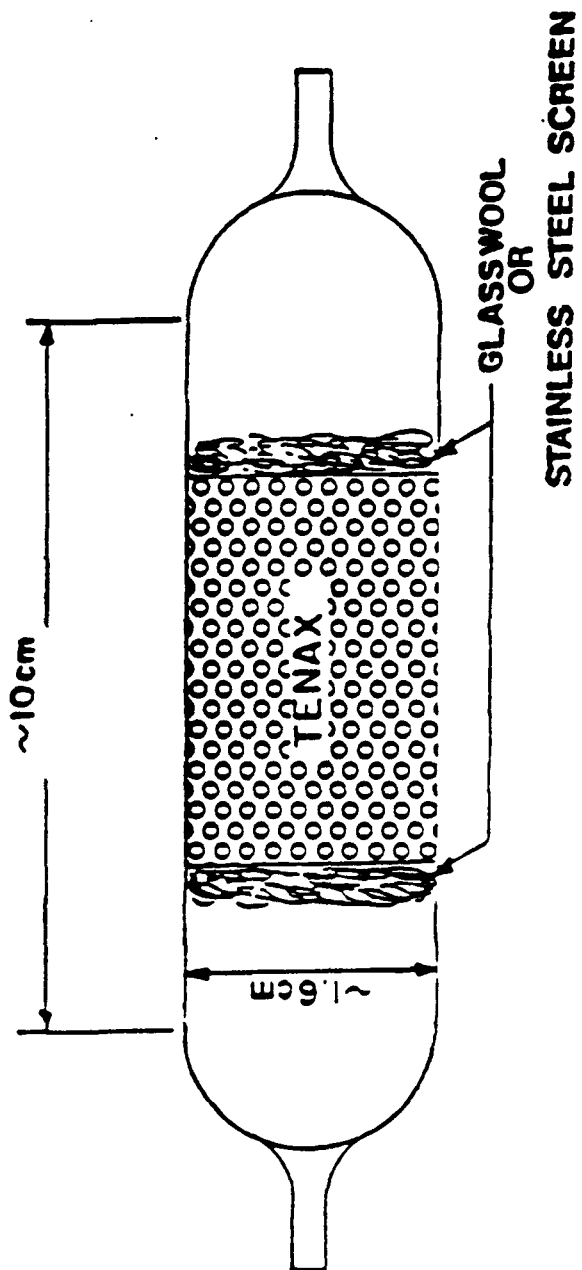


Figure 3. Inside-inside vial cartridge

All other sample transfer lines used with the VOST shall be Teflon with connecting fittings that are capable of forming leak-free, vacuum-tight connections without the use of sealing grease.

3.0 REAGENTS AND MATERIALS

3.1 2,6-Diphenylene oxide polymer (Tenax, 35/60 mesh):

3.1.1 The new Tenax is Soxhlet extracted for 24 hr with methanol (Burdick & Jackson, pesticide grade or equivalent). The Tenax is dried for 6 hr in a vacuum oven at 50°C before use. Users of I/O and I/I sorbent cartridges have used slightly different thermal conditioning procedures. I/O sorbent cartridges packed with Tenax are thermally conditioned by flowing organic-free nitrogen (30 mL/min) through the resin while heating to 190°C. Some users have extracted new Tenax and charcoal with pentane to remove nonpolar impurities. However, these users have experienced problems with residual pentane in the sorbents during analysis.

3.1.2 If very high concentrations of volatile POHCs have been collected on the resin (e.g., micrograms of analytes), the sorbent may require Soxhlet extraction as described above. Previously used Tenax cartridges are thermally reconditioned by the method described above.

3.2 Charcoal (SKC petroleum-base or equivalent): New charcoal is prepared and charcoal is reconditioned as described in Paragraph 4.4. New charcoal does not require treatment prior to assembly into sorbent cartridges. Users of VOST have restricted the types of charcoal used in sorbent cartridges to only petroleum-based types. Criteria for other types of charcoal are acceptable if recovery of POHC in laboratory evaluations meet the criteria of 50 to 150%.

3.3 Viton-O-Ring: All O-rings used in VOST shall be Viton. Prior to use, these O-rings should be thermally conditioned at 200°C for 48 hr. O-rings should be stored in clean, screw-capped glass containers prior to use.

3.4 Glass tubes/Condensers: The glass resin tubes and condensers should be cleaned with a nonionic detergent in an ultrasonic bath, rinsed well with organic-free water, and dried at 110°C. Resin tubes of the I/O design should be assembled prior to storage as described in Paragraph 4.1. Resin tubes of the I/I design can be stored in glass culture tube containers with cotton cushioning and Teflon-lined screw caps. Condensers can be capped with appropriate end caps prior to use.

3.5 Metal parts: The stainless steel carriers, C-clips, end plugs, and screens used in the I/O VOST design are cleaned by ultrasonication in a warm nonionic detergent solution, rinsed with distilled water, air-dried, and heated in a muffle furnace for 2 hr at 400°C. Resin tubes of the I/I design require Swagelok or equivalent end caps with Supelco M-1 ferrules. These should be heated at 190°C along with the assembled cartridges.

3.6 Silica gel (Indicating type, 6-16 mesh): New silica gel may be used as received. Silica gel which has been previously used should be dried for 2 hr at 175°C (350°F).

3.7 Cold packs: Any commercially available reusable liquids or gels that can be repeatedly frozen are acceptable. They are typically sold in plastic containers as "Blue Ice" or "Ice-Packs." Enough should be used to keep cartridges at or near 4°C.

3.8 Water: Water used for cooling train components in the field may be tap water; and water used for rinsing glassware should be organic-free.

3.9 Glass wool: Glass wool should be Soxhlet extracted for 8 to 16 hr, using methanol, and oven dried at 110°C before use.

4.0 SAMPLE HANDLING AND PROCEDURE

4.1 Assembly:

4.1.1 The assembly and packing of the sorbent cartridges should be carried out in an area free of volatile organic material, preferably a laboratory in which no organic solvents are handled or stored and in which the laboratory air is charcoal filtered. Alternatively, the assembly procedures can be conducted in a glove box which can be purged with organic-free nitrogen.

4.2 Tenax cartridges:

4.2.1 The Tenax, glass tubes, and metal cartridge parts are cleaned and stored (see Section 3.0). Approximately 1.6 g of Tenax is weighed and packed into the sorbent tube which has a stainless steel screen and C-clip (I/O design) or glass wool (I/I design) in the downstream end. The Tenax is held in place by inserting a stainless steel screen and C-clips in the upstream end (I/O design) or glass wool (I/I design). Each cartridge should be marked, using an engraving tool, with an arrow to indicate the direction of sample flow, and a serial number.

4.2.2 Conditioned resin tubes of the I/O design are then assembled into the metal carriers according to the previously described inside/inside or inside/outside procedures (with end caps) and are placed on cold packs for storage and transport. Conditioned resin tubes of the I/I design are capped and placed on cold packs for storage and transport.

4.3 Tenax/Charcoal tubes

4.3.1 The Tenax, charcoal, and metal cartridge parts are cleaned and stored as previously described (see Section 3.0). The tubes are packed with approximately a 3:1 volume ratio of Tenax and charcoal (approximately 1 g each). The Tenax and charcoal are held in place by the stainless steel screens and C-clips (I/O design) or by glass wool (I/I design). The glass tubes containing the Tenax and charcoal are then

conditioned as described below (see Paragraph 4.4). Place the I/O glass tubes in the metal carriers (see Paragraph 2.1.4.2.2), put end caps on the assembled cartridges, mark direction of sample flow and serial number, and place the assembled cartridges on cold packs for storage and transport.

4.3.2 Glass tubes of the I/I design are conditioned, and stored in the same manner as the I/O tubes.

4.4 Trap Conditioning - QC

4.4.1 Following assembly and leak-checking, the traps are connected in reverse direction to sampling to a source of organic-free nitrogen, and nitrogen is passed through each trap at a flow rate of 40 mL/min, while the traps are heated to 190°C for 12-28 hr. The actual conditioning period may be determined based on adequacy of the resulting blank checks.

4.4.2 The following procedure is used to blank check each set of sampling cartridges prior to sampling to ensure cleanliness. The procedure provides semi-quantitative data for organic compounds with boiling points below 110°C on Tenax and Tenax/Charcoal cartridges. It is not intended as a substitute for Method 5040.

4.4.2.1 The procedure is based on thermal desorption of each set of two cartridges, cryofocusing with liquid nitrogen onto a trap packed with glass beads, followed by thermal desorption from the trap and analysis by GC/FID.

4.4.2.2 The detection limit is based on the analysis of Tenax cartridges spiked with benzene and toluene and is around 2 ng for each compound.

4.4.2.3 The results of analyzing spiked cartridges on a daily basis should not vary by more than 20 percent. If the results are outside this range, the analytical system must be evaluated for the probable cause and a second spiked cartridge analyzed.

4.4.2.4 The GC operating conditions are as follows:

GC Operating Conditions

Column: Packed column 6 ft x 1/8" stainless steel 1.0 percent SP-1000 on Carbopack B 60/80, or equivalent.

Temperature program: 50°C for 5 min, 20°C/min increase to 190°C, hold 13 min.

Injector: 200°C.

Detector: F.I.D. 250°C.

Carrier Gas: Helium at 25 mL/min.

Sample valve: Valco 6-port with 40" x 1/16" stainless steel trap packed with 60/80 mesh glass beads.

Cryogen: Liquid nitrogen.

Trap heater: Boiling water, hot oil, or electrically heated.

Desorption heater: Supelco "clam shell" (high capacity carrier gas purifier) heater and Variac, adjusted to 180°C to 200°C.

4.4.2.5 Calibration

Accomplish calibration by analyzing a blank sorbent cartridge spiked with all TCL Volatile organics, surrogate spike compounds and internal standards.

Cartridge with benzene and toluene and analyzing according to standard operating procedure. A standard of benzene, toluene, and bromofluorobenzene (BFB) is prepared by injecting 2.0 uL of benzene and toluene and 1.0 uL of BFB into 10 mL of methanol. The concentration of this stock is 175 ng/uL of benzene and toluene, and 175 ng/uL BFB. One microliter of the stock standard is injected into a Tenax cartridge through a heated injection port set at 150°C. A GC oven can be used for this with the oven at room temperature. The carrier gas is at 50 mL/min. The solvent flush technique could be used. After the injection, remove the Tenax cartridge and place it in the desorption heater for analysis. BFB is also used as an internal standard spike for GC/MS analysis which provides a good comparison between GC/FID and GC/MS. The results of this spike analysis should not vary more than 5 percent day to day. Initially, then periodically, this spiked Tenax should be reanalyzed and time to verify that the 10 min desorption time and 180-200°C temperature are adequate to remove all of the spiked components. It should be noted that only one spiked Tenax cartridge need be prepared and analyzed daily unless otherwise needed to ensure proper instrument operation.

An acceptable blank level is left to the discretion of the analyst. An acceptable level is one that allows adequate quantitation of the analytes.

4.4.3 After conditioning, traps are sealed and placed on cold packs until sampling is accomplished. Conditioned traps should be held for a minimum amount of time to prevent the possibility of contamination.

4.4.4 It may be useful to spike the Tenax and Tenax/charcoal traps with the compounds of interest to ensure that they can be thermally desorbed under laboratory conditions. After spiked traps are analyzed they may be reconditioned and packed for sampling.

4.5.1 All trap components shall be assembled as previously described. A diagram of the trap assembly have been calibrated within 30 days prior to use.

The VOST is assembled according to the schematic diagram in Figure 1.

ATTACHMENT I

Sorbent Tube Specifications

Source: Method 0030, SW-846, Third Edition

2.1.4 Sorbent cartridges:

2.1.4.1 The sorbent cartridges used for the VOST may be used in either of two configurations: the inside-outside (I/O) configuration in which the cartridge is held within an outer glass tube and in a metal carrier, and the inside-inside (I/I) configuration in which only a single glass tube is used, with or without a metal carrier. In either case, the sorbent packing will be the same.

2.1.4.1.1 The first of a pair of sorbent cartridges shall be packed with approximately 1.6 g Tenax GC resin and the second cartridge of a pair shall be packed with Tenax GC and petroleum-based charcoal (3:1 by volume; approximately 1 g of each).

2.1.4.1.2 The second sorbent cartridge shall be packed so that the sample gas stream passes through the Tenax layer first and then through the charcoal layer.

2.1.4.2 The sorbent cartridges shall be glass tubes with approximate dimensions of 10 cm by 1.6 cm I.D. The two acceptable designs (I/O, I/I) for the sorbent cartridge are described in further detail below.

2.1.4.2.1 Inside/Inside sorbent cartridge: A diagram of an I/I sorbent cartridge is shown in Figure 3. This cartridge is a single glass tube (10 cm by 1.6 cm I.D.) which has the ends reduced in size to accommodate a 1/4- or 3/8-in. Swagelok or Cajon gas fitting. The resin is held in place by glass wool at each end of the resin layer. The amounts of each type of sorbent material used in the I/I design are the same as for the I/O design. Threaded end caps are placed on the sorbent cartridge after packing with sorbent to protect the sorbent from contamination during storage and transport.

2.1.4.2.2 Inside/Outside type sorbent cartridge: A diagram of an I/O sorbent cartridge is shown in Figure 4. In this design the sorbent materials are held in the glass tube with a fine mesh stainless steel screen and a C-clip. The glass tube is then placed within a larger diameter glass tube and held in place using Viton O-rings. The purpose of the outer glass tube is to protect the exterior of the resin-containing tube from contamination. The two glass tubes are held in a stainless steel cartridge holder, where the ends of the glass tubes are held in place by Viton O-rings placed in machine grooves in each metal end piece. The three cylindrical rods are secured in one of the metal end pieces and fastened to the other end piece using knurled nuts, thus sealing the glass tubes into the cartridge holder. The end pieces are fitted with a threaded nut onto which a threaded end cap is fitted with a Viton O-ring seal, to protect the resin from contamination during transport and storage.

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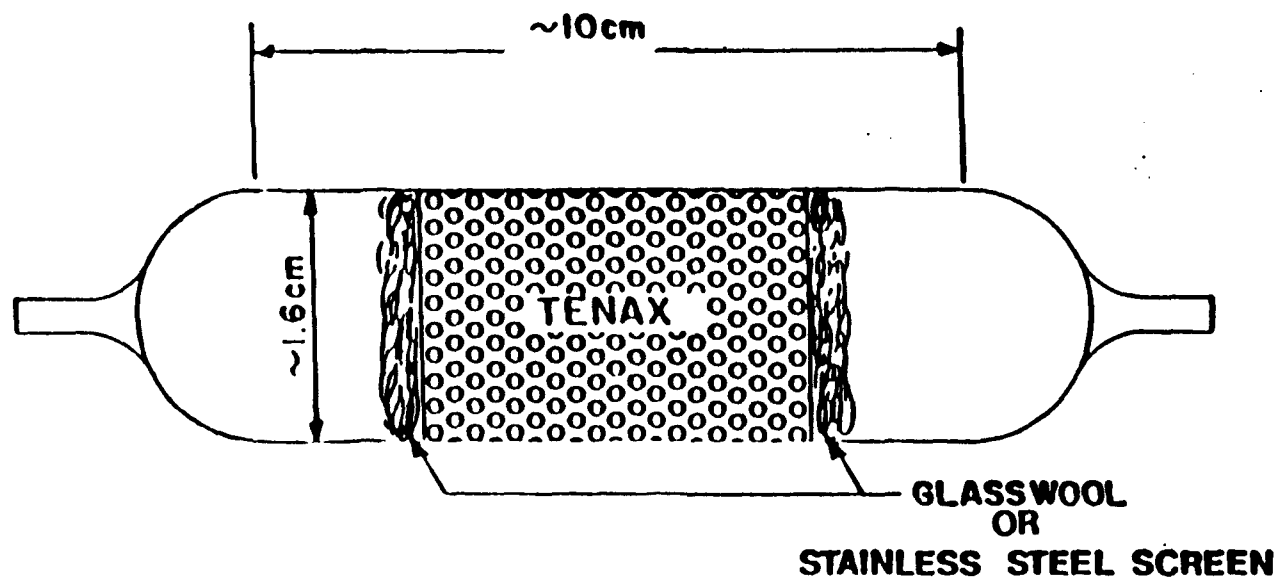


Figure 3. Inside-Inside vial cartridge

Revision 0
Date September 1986

All other sample transfer lines used with the VOST shall be Teflon with connecting fittings that are capable of forming leak-free, vacuum-tight connections without the use of sealing grease.

3.0 REAGENTS AND MATERIALS

3.1 2,6-Diphenylene oxide polymer (Tenax, 35/60 mesh):

3.1.1 The new Tenax is Soxhlet extracted for 24 hr with methanol (Burdick & Jackson, pesticide grade or equivalent). The Tenax is dried for 6 hr in a vacuum oven at 50°C before use. Users of I/O and I/I sorbent cartridges have used slightly different thermal conditioning procedures. I/O sorbent cartridges packed with Tenax are thermally conditioned by flowing organic-free nitrogen (30 mL/min) through the resin while heating to 190°C. Some users have extracted new Tenax and charcoal with pentane to remove nonpolar impurities. However, these users have experienced problems with residual pentane in the sorbents during analysis.

3.1.2 If very high concentrations of volatile POHCs have been collected on the resin (e.g., micrograms of analytes), the sorbent may require Soxhlet extraction as described above. Previously used Tenax cartridges are thermally reconditioned by the method described above.

3.2 Charcoal (SKC petroleum-base or equivalent): New charcoal is prepared and charcoal is reconditioned as described in Paragraph 4.4. New charcoal does not require treatment prior to assembly into sorbent cartridges. Users of VOST have restricted the types of charcoal used in sorbent cartridges to only petroleum-based types. Criteria for other types of charcoal are acceptable if recovery of POHC in laboratory evaluations meet the criteria of 50 to 150%.

3.3 Viton-O-Ring: All O-rings used in VOST shall be Viton. Prior to use, these O-rings should be thermally conditioned at 200°C for 48 hr. O-rings should be stored in clean, screw-capped glass containers prior to use.

3.4 Glass tubes/Condensers: The glass resin tubes and condensers should be cleaned with a nonionic detergent in an ultrasonic bath, rinsed well with organic-free water, and dried at 110°C. Resin tubes of the I/O design should be assembled prior to storage as described in Paragraph 4.1. Resin tubes of the I/I design can be stored in glass culture tube containers with cotton cushioning and Teflon-lined screw caps. Condensers can be capped with appropriate end caps prior to use.

3.5 Metal parts: The stainless steel carriers, C-clips, end plugs, and screens used in the I/O VOST design are cleaned by ultrasonication in a warm nonionic detergent solution, rinsed with distilled water, air-dried, and heated in a muffle furnace for 2 hr at 400°C. Resin tubes of the I/I design require Swagelok or equivalent end caps with Supelco M-1 ferrules. These should be heated at 190°C along with the assembled cartridges.

3.6 Silica gel (Indicating type, 6-16 mesh): New silica gel may be used as received. Silica gel which has been previously used should be dried for 2 hr at 175°C (350°F).

3.7 Cold packs: Any commercially available reusable liquids or gels that can be repeatedly frozen are acceptable. They are typically sold in plastic containers as "Blue Ice" or "Ice-Packs." Enough should be used to keep cartridges at or near 4°C.

3.8 Water: Water used for cooling train components in the field may be tap water; and water used for rinsing glassware should be organic-free.

3.9 Glass wool: Glass wool should be Soxhlet extracted for 8 to 16 hr, using methanol, and oven dried at 110°C before use.

4.0 SAMPLE HANDLING AND PROCEDURE

4.1 Assembly:

4.1.1 The assembly and packing of the sorbent cartridges should be carried out in an area free of volatile organic material, preferably a laboratory in which no organic solvents are handled or stored and in which the laboratory air is charcoal filtered. Alternatively, the assembly procedures can be conducted in a glove box which can be purged with organic-free nitrogen.

4.2 Tenax cartridges:

4.2.1 The Tenax, glass tubes, and metal cartridge parts are cleaned and stored (see Section 3.0). Approximately 1.6 g of Tenax is weighed and packed into the sorbent tube which has a stainless steel screen and C-clip (I/O design) or glass wool (I/I design) in the downstream end. The Tenax is held in place by inserting a stainless steel screen and C-clips in the upstream end (I/O design) or glass wool (I/I design). Each cartridge should be marked, using an engraving tool, with an arrow to indicate the direction of sample flow, and a serial number.

4.2.2 Conditioned resin tubes of the I/O design are then assembled into the metal carriers according to the previously described inside/inside or inside/outside procedures (with end caps) and are placed on cold packs for storage and transport. Conditioned resin tubes of the I/I design are capped and placed on cold packs for storage and transport.

4.3 Tenax/Charcoal tubes

4.3.1 The Tenax, charcoal, and metal cartridge parts are cleaned and stored as previously described (see Section 3.0). The tubes are packed with approximately a 3:1 volume ratio of Tenax and charcoal (approximately 1 g each). The Tenax and charcoal are held in place by the stainless steel screens and C-clips (I/O design) or by glass wool (I/I design). The glass tubes containing the Tenax and charcoal are then

conditioned as described below (see Paragraph 4.4). Place the I/O glass tubes in the metal carriers (see Paragraph 2.1.4.2.2), put end caps on the assembled cartridges, mark direction of sample flow and serial number, and place the assembled cartridges on cold packs for storage and transport.

4.3.2 Glass tubes of the I/I design are conditioned, and stored in the same manner as the I/O tubes.

4.4 Trap Conditioning - QC

4.4.1 Following assembly and leak-checking, the traps are connected in reverse direction to sampling to a source of organic-free nitrogen, and nitrogen is passed through each trap at a flow rate of 40 mL/min, while the traps are heated to 190°C for 12-28 hr. The actual conditioning period may be determined based on adequacy of the resulting blank checks.

4.4.2 The following procedure is used to blank check each set of sampling cartridges prior to sampling to ensure cleanliness. The procedure provides semi-quantitative data for organic compounds with boiling points below 110°C on Tenax and Tenax/Charcoal cartridges. It is not intended as a substitute for Method 5040.

4.4.2.1 The procedure is based on thermal desorption of each set of two cartridges, cryofocusing with liquid nitrogen onto a trap packed with glass beads, followed by thermal desorption from the trap and analysis by GC/FID.

4.4.2.2 The detection limit is based on the analysis of Tenax cartridges spiked with benzene and toluene and is around 2 ng for each compound.

4.4.2.3 The results of analyzing spiked cartridges on a daily basis should not vary by more than 20 percent. If the results are outside this range, the analytical system must be evaluated for the probable cause and a second spiked cartridge analyzed.

4.4.2.4 The GC operating conditions are as follows:

GC Operating Conditions

Column: Packed column 6 ft x 1/8" stainless steel 1.0 percent SP-1000 on Carbopack B 60/80, or equivalent.

Temperature program: 50°C for 5 min, 20°C/min increase to 190°C, hold 13 min.

Injector: 200°C.

Detector: F.I.D. 250°C.

Carrier Gas: Helium at 25 mL/min.

Sample valve: Valco 6-port with 40" x 1/16" stainless steel trap packed with 60/80 mesh glass beads.

Cryogen: Liquid nitrogen.

Trap heater: Boiling water, hot oil, or electrically heated.

Desorption heater: Supelco "clam shell" (high capacity carrier gas purifier) heater and Variac, adjusted to 180°C to 200°C.

4.4.2.5 Calibration

Accomplish calibration by analyzing a blank sorbent cartridge spiked with all TCL Volatile organics, surrogate spike compounds and internal standards.

Cartridge with benzene and toluene and analyzing according to standard operating procedure. A standard of benzene, toluene, and bromofluorobenzene (BFB) is prepared by injecting 2.0 uL of benzene and toluene and 1.0 uL of BFB into 10 mL of methanol. The concentration of this stock is 175 ng/uL of benzene and toluene, and 175 ng/uL BFB. One microliter of the stock standard is injected into a Tenax cartridge through a heated injection port set at 150°C. The oven can be used for this with the oven at room temperature. The carrier gas is at 50 mL/min. The solvent flush technique should be used. After the injection, remove the Tenax cartridge and place it in the desorption heater for analysis. BFB is also used as an internal standard spike for GC/MS analysis which provides a good comparison between GC/FID and GC/MS. The results of this spike analysis should not vary more than 5 percent day to day. Initially, then periodically, this spiked Tenax should be reanalyzed and time to verify that the 10 min desorption time and 180-200°C temperature are adequate to remove all of the spiked components. It should be noted that only one spiked Tenax cartridge need be prepared and analyzed daily unless otherwise needed to ensure proper instrument operation.

An acceptable blank level is left to the discretion of the analyst. An acceptable level is one that allows adequate detection of the components of interest.

4.4.3 After conditioning, traps are sealed and placed on cold packs until sampling is accomplished. Conditioned traps should be held for a minimum amount of time to prevent the possibility of contamination.

4.4.4 It may be useful to spike the Tenax and Tenax/charcoal traps with the compounds of interest to ensure that they can be thermally desorbed under laboratory conditions. After spiked traps are analyzed they may be reconditioned and packed for sampling.

4.5.1 All trap components shall be assembled as previously described. A trap shall have been calibrated within 30 days prior to use.

The VOST is assembled according to the schematic diagram in

ATTACHMENT II

SORBENT TUBE SAS

I. PERFORM THE FOLLOWING PER CLP STATEMENT OF WORK, CURRENT SOW

1. GC/MS Tuning
2. Qualitative Compound Identification
3. Surrogate Spike (compounds to be used and their concentrations as they are specified in the SOW) to each standards, blanks, and samples. The concentration of each surrogate spike compound shall be 200 total ng.
4. Internal Standard Compounds to be added at 200 total ng each.
5. For matrix spike/matrix spike duplicate analysis, matrix spike compounds specified in the CLP Statement of Work shall be used. The total concentration of each compound shall be 200 ng.
6. Tentatively identified of non-target compound (TICs).

II. CALIBRATION REQUIREMENTS

1. Initial Calibration

Initial calibration curve shall consist of 5 points at 20, 200, 500, 750, and 1,000 total ng of each compound.
2. Continuing Calibration Check
 - a. Perform daily continuing calibration check at the beginning before sample analysis, and every 12-hour shift using standards at a concentration of 20 ng.
 - b. Perform weekly using standard at concentration of 20 ng.

III. PREPARATION AND ANALYSIS OF STANDARDS

Calibration standards shall be prepared by spiking pre-cleaned blank cartridges with appropriate amount of standards, and analyzed the same way as the samples.

- ° Extra tubes must be prepared by the laboratory and sent to the field for the MS/MSD analyses. Tubes will be labeled for the MS/MSD analyses; these will be blank tubes resubmitted from the field. If no field samples are designated for MS/MSD by field staff, laboratory shall prepare control spikes consisting of duplicate spike blank cartridges.

IV. DETECTION LIMIT VERIFICATION

Spike a blank Tenax-Tenax/charcoal cartridge pair with all target compounds at the required quantitation limits as listed in Table 1. All qualitative identification criteria as per the CLP Statement of Work (SOW) for organics shall be met. Provide all documentation requirements of the CLP organic SOW.

V. ANALYSIS

Analyze and report results of each cartridge pair in the unit of ng/tube, and convert to $\mu\text{g}/\text{m}^3$ (Attachment III, page III-1).

TABLE 1

TARGET COMPOUND LIST (TCL) AND QUANTITATION LIMITS (QLs)

<u>Volatile Organics</u>	<u>CAS Number</u>	<u>Required Quantitation Limits (ng/tube)</u>
Benzene	71-43-2	5
Bromodichloromethane	75-27-4	5
Bromoform	75-25-2	5
Bromomethane	74-83-9	5
Carbon Tetrachloride	56-23-5	5
Chlorobenzene	108-90-7	5
Chloroethane	75-00-3	5
Chloroform	67-66-3	5
Chloromethane	74-87-3	5
Dibromochloromethane	124-48-1	5
1,1-Dichloroethane	75-34-3	5
1,2-Dichloroethane	107-06-2	5
1,1-Dichloroethene	75-35-4	5
1,2-Dichloroethene (Total)		5
1,2-Dichloropropene	78-87-5	5
Cis-1,3-Dichloropropene	10061-01-5	5
Trans-1,3-Dichloropropene	10061-02-6	5
Ethyl Benzene	100-51-4	5
Methylene Chloride	75-09-2	5
1,1,2,2-Tetrachloroethane	79-34-5	5
Tetrachloroethene	127-18-4	5
Toluene	108-88-3	5
1,1,1-Trichloroethane	71-55-6	5
1,1,2-Trichloroethane	79-00-5	5
Trichloroethene	79-01-6	5
Vinyl Chloride	75-01-4	5
Acetone	67-64-1	5
Carbon Disulfide	75-15-0	5
2-Butanone	78-93-3	5
Vinyl Acetate	108-05-4	5
4-Methyl-2-Pentanone	108-10-1	5
2-Hexanone	519-78-6	5
Styrene	100-42-5	5
m-Xylene**	108-38-3	5
o-Xylene**	95-47-6	5
p-Xylene**	106-42-3	5

** Assuming cartridge is packed with 2 grams of Tenax.

A/P/HIMCO/AD4

ATTACHMENT III

SORBENT TUBE SAS ADDITIONAL SPECIAL INSTRUCTIONS

Daily GC/MS performance test, Section 9, will be performed by adding 50 ng of bromofluorobenzene to a blank Tenax cartridge.

Internal Standard Calibration, Section 6.2, 50 ng of bromofluorobenzene (BFB) will be added to all sorbent cartridges (in addition to one or more internal standards) to provide continuous monitoring of the GC/MS performance relative to BFB.

The laboratory will tentatively identify up to 10 additional compounds.

For MS/MSD analyses, the Tenax/charcoal tube must be spiked if the analysis is run with the tubes in series. The sampling team may request the tubes not be run in series if field screening indicates high concentrations.

Surrogate spikes will be comprised of the following:

- ° D₈ - Toluene
- ° D₄ - 1,2-dichloroethene
- ° 4 - bromofluorobenzene

These surrogate compounds will go into the Tenax/charcoal tube if the tubes are to be analyzed in pairs. If the Tenax and Tenax/charcoal tubes are analyzed separately, each tube must be spiked with the surrogates. Tubes will be spiked with 200 ng of each surrogate compounds.

DATA CALCULATION AND REPORTING/UNITS

Data is calculated in units of ng/tube (Section 7.5.1.1, page 5040-7). Units of ng/tube should be converted to ng/L to indicate the ng of each volatile organic compound detected (or undetected) per liter of soil gas collected at standard conditions, using the following conversion:

$$\text{ng/tube} \times \frac{\text{tube}}{\text{Liters}^*} = \text{ng/Liter}$$

*Supplied by field team, corrected to standard conditions will be recorded on chain-of-custody form.

ATTACHMENT IV

SORBENT TUBE SAS ANALYTICAL RESULTS REQUIRED

- All documentation requirements of the CLP organic SOW 2/88 applies, including copies of the chain-of-custody forms received at the lab with the samples, initial and continuing calibration forms, volatile OADS, surrogate recovery summary, internal standard summary, and matrix, matrix spike duplicate recovery summary.
- Non-target list compounds detected will be identified, quantified, and reported as TICs in the CLP 2/88 SOW.
- All results from analysis of blank cartridges.
- Detection limit verification data from procedure in Attachment II.
- Notes on calculation of percent rsd for calibration curve and percent rpd for daily calibration check standards.
- Documentation of cartridge tube pairs preparation and handling.

A/P/HIMCO/AD4

PROTOCOL FOR ANALYSIS OF SORBENT CARTRIDGES
FROM VOLATILE ORGANIC SAMPLING TRAIN

1.0 SCOPE AND APPLICATION

1.1 Method 5040 was formerly Method 3720 in the Second Edition of this manual.

1.2 This method covers the determination of volatile principal organic hazardous constituents (POHCs), collected on Tenax and Tenax/charcoal sorbent cartridges using a volatile organic sampling train, VOST (1). Much of the description for purge-and-trap GC/MS analysis is described in Method 8240 of this chapter. Because the majority of gas streams sampled using VOST will contain a high concentration of water, the analytical method is based on the quantitative thermal desorption of volatile POHCs from the Tenax and Tenax/charcoal traps and analysis by purge-and-trap GC/MS. For the purposes of definition, volatile POHCs are those POHCs with boiling points less than 100°C.

1.3 This method is applicable to the analysis of Tenax and Tenax/charcoal cartridges used to collect volatile POHCs from wet stack gas effluents from hazardous waste incinerators.

1.4 The sensitivity of the analytical method for a particular volatile POHC depends on the level of interferences and the presence of detectable levels of volatile POHCs in blanks. The desired target detection limit of the analytical method is 0.1 ng/L (20 ng on a single pair of traps) for a particular volatile POHC desorbed from either a single pair of Tenax and Tenax/charcoal cartridges or by thermal desorption of up to six pairs of traps onto a single pair of Tenax and Tenax/charcoal traps. The resulting single pair of traps is then thermally desorbed and analyzed by purge-and-trap GC/MS.

1.5 This method is recommended for use only by experienced mass spectroscopists or under the close supervision of such qualified persons.

2.0 SUMMARY OF METHOD

2.1 A schematic diagram of the analytical system is shown in Figure 1. The contents of the sorbent cartridges are spiked with an internal standard and thermally desorbed for 10 min at 180°C with organic-free nitrogen or helium gas (at a flow rate of 40 mL/min), bubbled through 5 mL of organic-free water, and trapped on an analytical adsorbent trap. After the 10-min desorption, the analytical adsorbent trap is rapidly heated to 180°C, with the carrier gas flow reversed so that the effluent flow from the analytical trap is directed into the GC/MS. The volatile POHCs are separated by temperature-programmed gas chromatography and detected by low-resolution mass spectrometry. The concentrations of volatile POHCs are calculated using the internal standard technique.

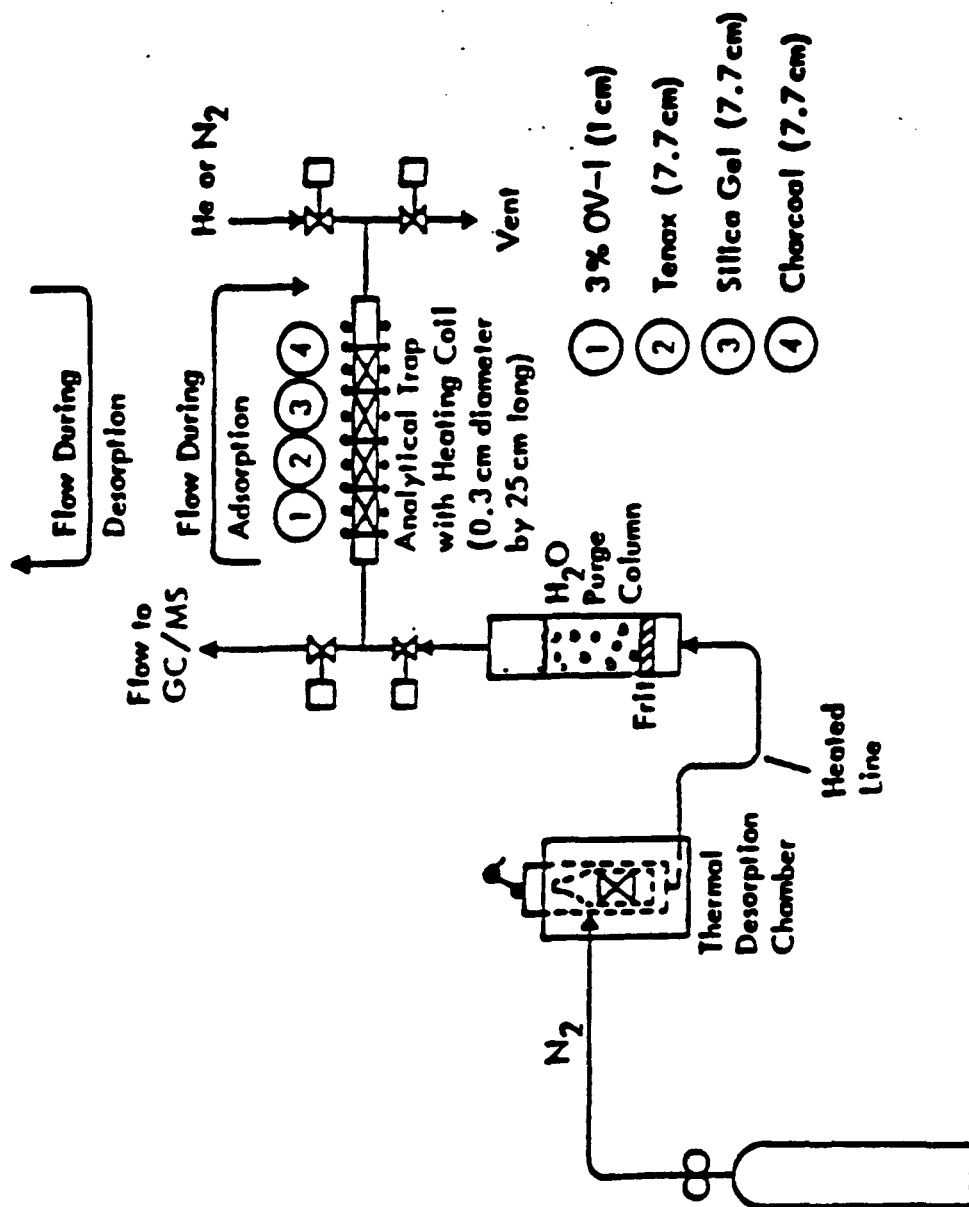


Figure 1. Schematic diagram of trap desorption/analysis system.

3.0 INTERFERENCES

3.1 Refer to Methods 3500 and 8240.

4.0 APPARATUS AND MATERIALS

4.1 Thermal desorption unit:

4.1.1 The thermal desorption unit (for Inside/Inside VOST cartridges, use Supelco "clamshell" heater; for Inside/Outside VOST cartridges, user fabricated unit is required) should be capable of thermally desorbing the sorbent resin tubes. It should also be capable of heating the tubes to $180 \pm 10^{\circ}\text{C}$ with flow of organic-free nitrogen or helium through the tubes.

4.2 Purge-and-trap unit:

4.2.1 The purge-and-trap unit consists of three separate pieces of equipment: the sample purger, trap, and the desorber. It should be capable of meeting all requirements of Method 5030 for analysis of purgeable organic compounds from water.

4.3 GC/MS system: As described in Method 8240.

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as water in which an interferent is not observed at the method detection limit of the parameters of interest.

5.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 450 g of activated carbon (Calgon Corporation, Filtrasorb-300, or equivalent).

5.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

5.1.3 Reagent water may also be prepared by boiling distilled water for 15 min. Subsequently, while maintaining the temperature at 90°C , bubble a contaminant-free inert gas through the water for 1 hr. Allow the water to cool to room temperature while continuing to bubble the inert gas through the water. This water should be transferred directly to the purge-and-trap apparatus for use.

5.1.4 Other methods that can be shown to produce organic-free water can be used.

5.2 Analytical grade reagents:

5.2.1 2,6-Diphenylene oxide polymer: Tenax (60/80 mesh), chromatographic grade or equivalent.

5.2.2 Methyl silicone packing: 3% OV-1 on Chromosorb W (60/80 mesh) or equivalent.

5.2.3 Silica gel: Davison Chemical (35/00 mesh), Grade 15, or equivalent.

5.2.4 Charcoal: Petroleum-based (SKC Lot 104 or equivalent).

5.3 Stock standard solution:

5.3.1 Stock standard solutions will be prepared from pure standard materials or purchased as certified solutions. The stock standards should be prepared in methanol using assayed liquids or gases, as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA-approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

5.3.2 Fresh stock standards should be prepared weekly for volatile POHCs with boiling points of $<35^{\circ}\text{C}$. All other standards must be replaced monthly, or sooner if comparison with check standards indicates a problem.

5.4 Secondary dilution standards:

5.4.1 Using stock standard solutions, prepare in methanol secondary dilution standards that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the desorbed calibration standards will bracket the working range of the analytical system.

5.5 4-Bromofluorobenzene (BFB) standard:

5.5.1 Prepare a 25 ng/uL solution of BFB in methanol.

Use CLP
surrogates:

D₄-1,2-dichloroethene, D₈-toluene

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to Method 0030, Chapter Ten.

6.2 ^{Cartridges}
~~Sample trains~~ obtained from the VOST should be analyzed within
~~2-6 weeks~~ 7 days of sample collection.

7.0 PROCEDURE

7.1 Assembly of PTD device:

7.1.1 Assemble a purge-and-trap desorption device (PTD) that meets all the requirements of Method 5030 (refer to Figure 1).

7.1.2 Connect the thermal desorption device to the PTD device. Calibrate the PTD-GC/MS system using the internal standard technique.

7.2 Internal standard calibration procedure:

7.2.1 This approach requires the use of deuterated benzene as the internal standard for these analyses. Other internal standards may be proposed for use in certain situations. The important criteria for choosing a particular compound as an internal standard are that it be similar in analytical behavior to the compounds of interest and that it can be demonstrated that the measurement of the internal standard be unaffected by method or matrix interferences. Other internal standards that have been used are d_{10} -ethylbenzene and d_4 -1,2-dichloroethane. One adds 50 ng of BFB to all sorbent cartridges (in addition to one or more internal standards) to provide continuous monitoring of the GC/MS performance relative to BFB.

7.2.2 Prepare calibration standards at a minimum of ^{FIVE} ~~three~~ concentration levels for each analyte of interest.

7.2.3 The calibration standards are prepared by spiking a blank Tenax or Tenax/charcoal trap with a methanolic solution of the calibration standards (including 50 ng of the internal standard, such as deuterated benzene), using the flash evaporation technique. The flash evaporation technique requires filling the needle of a 5.0- μ L syringe with clean methanol and drawing air into the syringe to the 1.0- μ L mark. This is followed by drawing a methanolic solution of the calibration standards (containing 25 μ g/ μ L of the internal standard) to the 2.0- μ L mark. The glass traps should be attached to the injection port of a gas chromatograph while maintaining the injector temperature at 160°C. The carrier gas flow through the traps should be maintained at about 50 mL/min.

7.2.4 After directing the gas flow through the trap, the contents of the syringe should be slowly expelled through the gas chromatograph injection port over about 15 sec. After 25 sec have elapsed, the gas flow through the trap should be shut off, the syringe removed, and the trap analyzed by the PTD-GC/MS procedure outlined in Method 8240. The total flow of gas through the traps during addition of calibration standards to blank cartridges, or internal standards to sample cartridges, should be 25 mL or less.

7.2.5 Analyze each calibration standard for both Tenax and Tenax/charcoal cartridges according to Section 7.3. Tabulate the area response

of the characteristic ions of each analyte against the concentration of the internal standard and calculate response factor (RF) for each compound, using Equation 1.

$$RF = A_S C_{IS} / A_{IS} C_S \quad (1)$$

where:

A_S = Area of the characteristic ion for the analyte to be measured.

A_{IS} = Area of the characteristic ion for the internal standard.

C_{IS} = Amount (ng) of the internal standard.

C_S = Amount (ng) of the volatile POHC in calibration standard.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant, and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_S/A_{IS} versus RF.

7.2.6 The working calibration curve or RF must be verified on each working day by the measurement of one or more of the calibration standards. If the response varies by more than +25% for any analyte, a new calibration standard must be prepared and analyzed, for that analyte.

7.3 The schematic of the PTD-GC/MS system is shown in Figure 1. The sample cartridge is placed in the thermal desorption apparatus (for Inside/Inside VOST cartridges, use Supelco "clamshell" heater; for Inside/Outside VOST cartridges, user fabricated unit is required) and desorbed in the purge-and-trap system by heating to 180°C for 10 min at a flow rate of 40 mL/min. The desorbed components pass into the bottom of the water column, are purged from the water, and collected on the analytical adsorbent trap. After the 10-min desorption period, the compounds are desorbed from the analytical adsorbent trap into the GC/MS system according to the procedures described in Method 8240.

7.4 Qualitative identification:

7.4.1 The procedure for qualitative identification of volatile POHCs using this protocol is described in Method 8240.

7.5 Calculations:

7.5.1 When an analyte has been qualitatively identified, quantification should be based on the integrated abundance from the EICP of the primary characteristic ion chosen for that analyte. If the sample produces an interference for the primary characteristic ion, a secondary characteristic ion should be used.

7.5.1.1 Using the internal standard calibration procedure, the amount of analyte in the sample cartridge is calculated using the response factor (RF) determined in Paragraph 7.2.5 and Equation 2.

$$\text{Amount of POHC} = A_s C_{is} / A_{is} RF \quad (2)$$

where:

A_s = Area of the characteristic ion for the analyte to be measured.

A_{is} = Area for the characteristic ion of the internal standard.

C_{is} = Amount (ng) of internal standard.

7.5.1.2 The amount of POHCs in a sample cartridge collected by VOST for incinerator trial burns is a regulatory determination. Procedures used extensively by one user are outlined below.

7.5.1.3 The total amount of the POHCs of interest collected in a pair of traps should be summed. These values should then be blank corrected. Guidelines for blank correction of sample cartridges are outlined below.

7.5.1.3.1 After all blanks (field and trip) are analyzed, a paired t-test should be used to determine whether trip blanks are significantly different from field blanks. If no difference is found, then the mean and standard deviation of the combined field and trip blanks for each POHC of interest is calculated.

7.5.1.3.2 If, when using the paired t-test, the field and trip blanks are determined to be different, then the field blank (or the mean of multiple field blanks) associated with a particular run should be used as the blank value for that particular run.

7.5.1.4 Next, for each sample/POHC combination, a determination must be made as to whether a particular sample is significantly different from the associated blank. If the mean of trip and field blanks is used, then a sample is different from blank if:

$$(\text{measured sample value}) - (\text{mean blank value}) > (3 \times \text{blank standard deviation})$$

(If an individual field blank is used as the blank value, the above criteria do not apply.) If the sample is determined to be different from the blank according to the above criteria, then the amount of a particular POHC is blank-corrected by subtracting the blank value from the sample value.

disregard

7.5.1.5 If, according to the above procedure, the sample cannot be distinguished from the blank (i.e., for a given run, there is a high sample value and high blank value or there is a low sample value and low blank value), the measured sample value is used as the blank-corrected value. In this case, the measured sample value is used to calculate the maximum emission value (and therefore a minimum DE value) for a particular run.

disregard

7.5.1.6 The observation of high concentrations of POHCs of interest in blank cartridges indicates possible residual contamination of the sorbent cartridges prior to shipment to and use at the site. Data that fall in this category (especially data indicating high concentrations of POHCs in blank sorbent cartridges) should be qualified with respect to validity, and blank data should be reported separately. The applicability of data of this type to the determination of a site is a regulatory decision. Continued observation of high concentrations of POHCs in blank sorbent cartridges indicates that procedures for cleanup, monitoring, shipment, and storage of sorbent cartridges by a particular user be investigated to eliminate this problem.

7.5.1.7 If any internal standard recoveries fall outside the limits established in Section 8.4, data for all analytes determined for that cartridge(s) must be qualified.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 3500 for sample preparation procedures.

8.2 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of blank Tenax and Tenax/charcoal cartridges spiked with the analytes of interest. The laboratory is required to maintain performance records to define the quality of data that are generated. Ongoing performance checks must be compared with established performance criteria to determine if results are within the expected precision and accuracy limits of the method.

8.2.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable precision and accuracy with this method. This ability is established as described in Paragraph 7.2.

8.2.2 The laboratory must spike all Tenax and Tenax/charcoal cartridges with the internal standard(s) to monitor continuing laboratory performance. This procedure is described in Paragraph 7.2.

8.3 To establish the ability to generate acceptable accuracy and precision, the analyst must spike blank Tenax and Tenax/charcoal cartridges with the analytes of interest at two concentrations in the working range.

8.3.1 The average response factor (R) and the standard deviation (S) for each must be calculated.

8.3.2 The average recovery and standard deviation must fall within the expected range for determination of volatile POHCs using this method. The expected range for recovery of volatile POHCs using this method is 50-150%.

8.4 The analyst must calculate method performance criteria for the internal standard(s).

8.4.1 Calculate upper and lower control limits for method performances using the average area response (A) and standard deviation(s) for internal standard:

Upper Control Limit (UCL) = $A + 3S$.

Lower Control Limit (LCL) = $A - 3S$.

The UCL and LCL can be used to construct control charts that are useful in observing trends in performance. The control limits must be replaced by method performance criteria as they become available from the U.S. EPA.

8.5 The laboratory is required to spike all sample cartridges (Tenax and Tenax/charcoal) with internal standard.

8.6 Each day, the analyst must demonstrate through analysis of blank Tenax and Tenax/charcoal cartridges and reagent water that interferences from the analytical system are under control.

8.7 The daily GC/MS performance tests required for this method are described in Method 8240.

9.0 METHOD PERFORMANCE

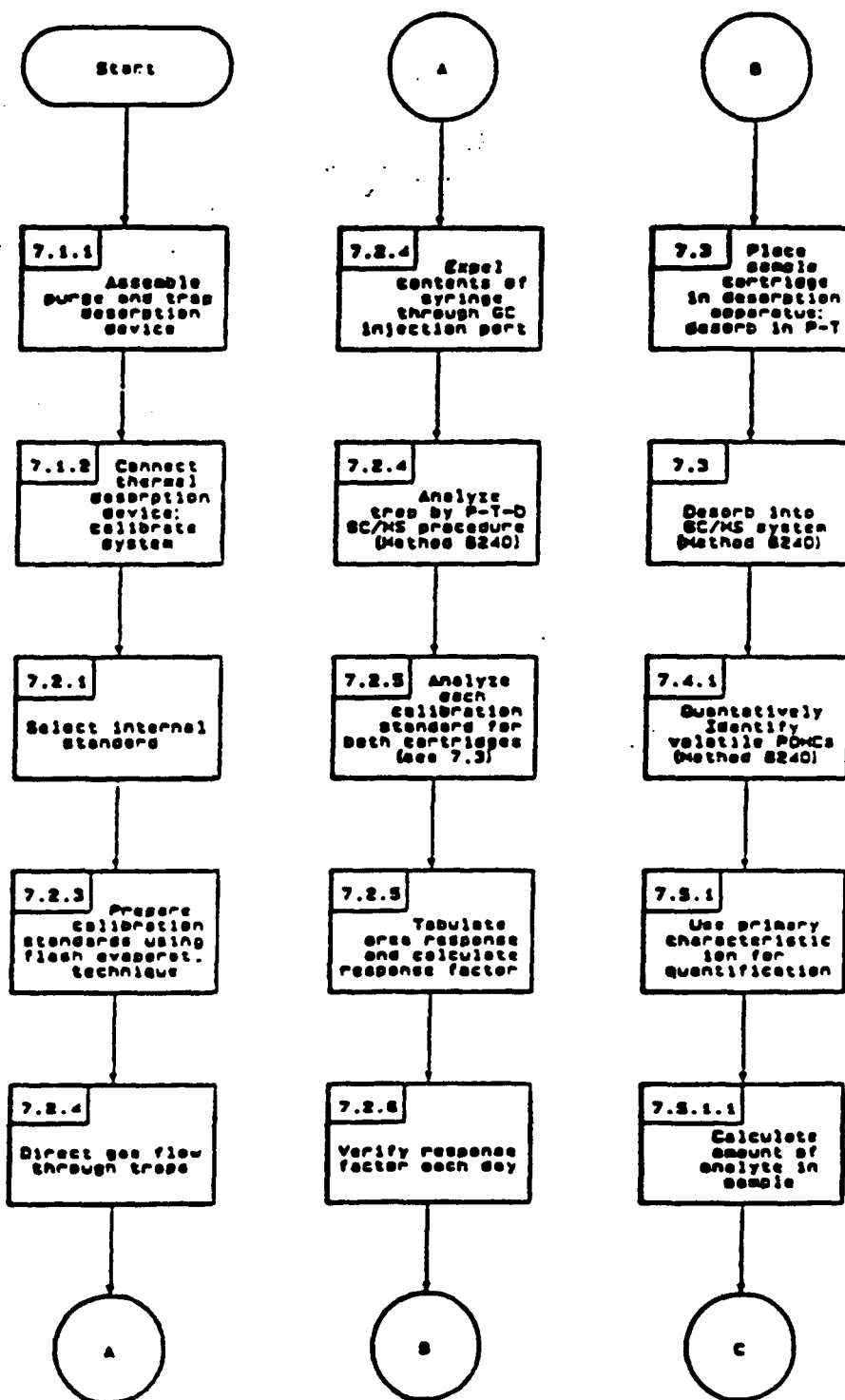
9.1 Refer to the determinative methods for performance data.

10.0 REFERENCES

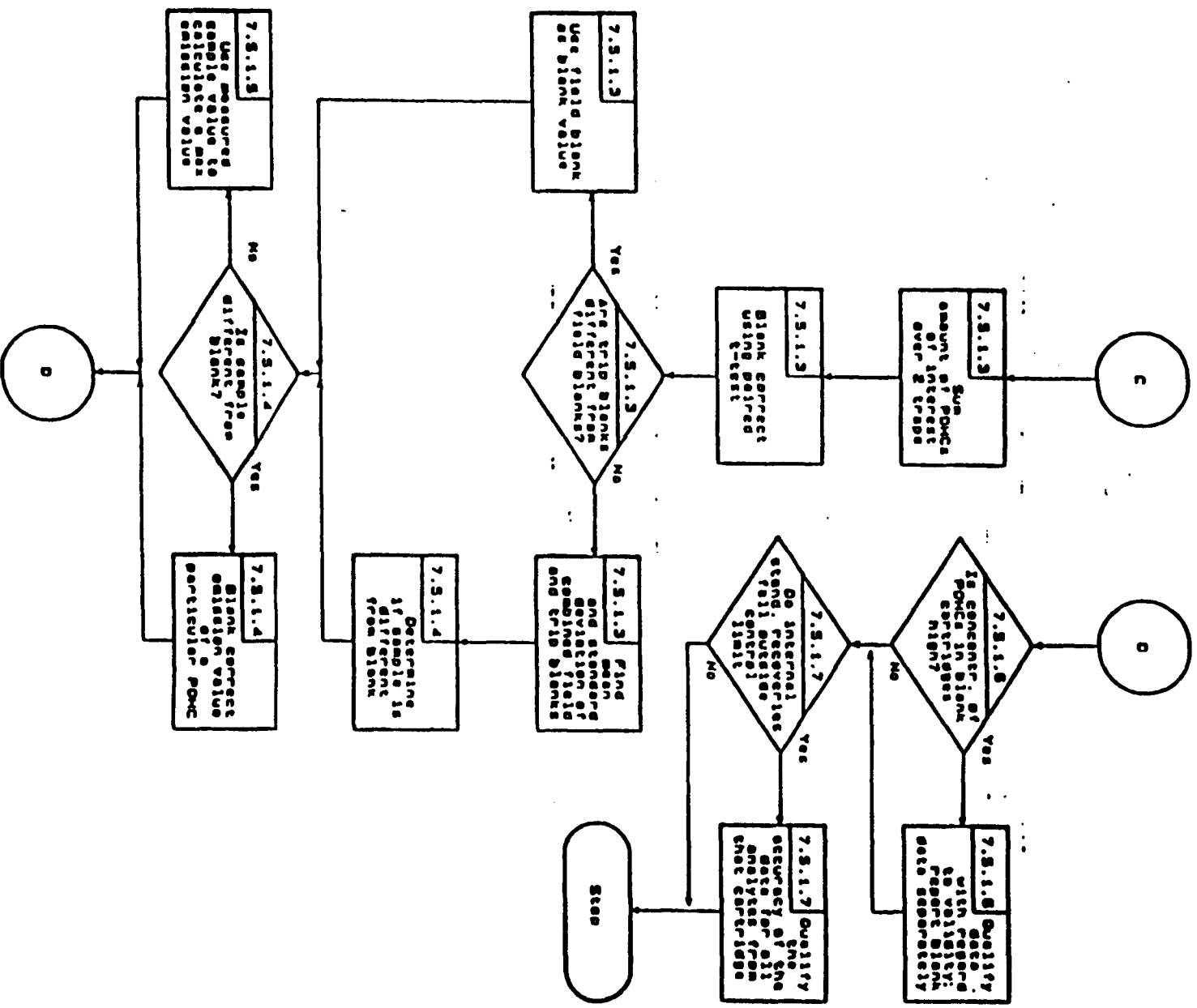
1. Protocol for Collection and Analysis of Volatile POHC's Using VOST. EPA/600/8-84-007, March 1984.

2. Validation of the Volatile Organic Sampling Train (VOST) Protocol. Volumes I and II. EPA/600/4-86-014a, January 1986.

METHOD 5040
 PROTOCOL FOR ANALYSIS OF SORBENT CARTRIDGES
 FROM VOLATILE ORGANIC SAMPLING TRAIN



METHOD 5040
 PROTOCOL FOR ANALYSIS OF SORBENT CARTRIDGES
 FROM VOLATILE ORGANIC SAMPLING TRAIN
 (Continued)



5040 - 11

Revision 0
 Date September 1986

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request



Regional Transmittal



Telephone Request

- A. EPA Region/Client: — V —
- B. RSCC Representative: Jan Pels
- C. Telephone Number: (312) 353-2720
- D. Date of Request: _____
- E. Site Name: Himco Dump, Elkhart Indiana

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: _____
physical soil tests: Atterberg Limits
Particle size analysis
permeability, consolidation, triaxial shear
2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):
25 low level soils for Atterberg limits + grain size
3 low level soils for permeability
2 low level soils for consolidation
5 low level soils for triaxial shear
3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):
Superfund remedial investigation

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: _____

overnight courier - expect one shipment for
Atterberg particle size Shelby tubes for consolidation,
triaxial shear + permeability in another shipment

6. Number of days analysis and data required after laboratory receipt of samples:
30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): Particle size: ASTM D422, D421, D854
Atterberg limits: ASTM D4318 (attached)

Permeability: ASTM D2434 (attached) +

Consolidation: ASTM D2435 (attached) Triaxial Shear: ASTM D2850 (attached)

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): see Attachment 1 (particle size)

Use only the methods specified above. Obtain approval
of Region I prior to use of any other method.

Rebound/unloading characteristics for consolidation not
necessary, use at least 3 load increments in establishing
time-deformation curve. Triaxial: use at least 10 readings
to define the stress-strain curve.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.

see attached, Attachment 2 (particle size)

10. Other (use additional sheets or attach supplementary information, as needed):
Lab data will be rejected and non-payment will be recommended
if lab does not follow methods referenced above.

11. Name of sampling/shipping contact: Marcia Kuenl

Phone: (414) 458-8711 x2253

8. cont. Sieve, hydrometer, specific gravity and hygroscopic moisture analysis
MUST be performed. Absolutely NO OTHER METHODOLOGY MAY BE USED
unless lab has obtained approval of SMO prior to analysis.

8. cont. use ASTM D2434 permeability method for samples
received in ziploc bags (disturbed) that will contain
granular soil

use Falling head method for samples received in
Shelby tubes that will c-6 contain clay/silt, need
complete saturation

9. Analytical Results Required

Grain Size - see Attachment 2

- raw data including container tare weights
- report results as % finer than the specified particle diameter
- grain size distribution curve on semilog paper: % finer by weight on arithmetic scale, grain size on log scale

Atterberg Limits

- all raw data sheets
- report results as:
liquid limit, plastic limit & plasticity index to nearest whole number

Consolidation

- preparation of soil, trimming procedure
- initial & final moisture content
- dry mass, initial & final wet unit weight
- initial percent saturation
- specimen dimensions
- condition of test
- plot of log of time or square root of time vs. deformation
- plot of void ratio vs. log of pressure curve or percent compression vs. log of pressure curve
- plot of log of coefficient vs. log of average pressure

9. Analytical Results (cont.)

Triaxial Shear

- specimen description
- initial dry unit weight - water content, when water content obtained
- degree of saturation
- height - diameter of specimen, height to diameter ratio
- value of compressive strength, values of minor + major principal stresses at failure
- stress-strain curve
- axial strain at failure (percent)
- average rate of axial strain to failure (percent per minute)
- sketch or photo showing type of failure
- membrane correction equation used (if applicable)

Permeability - Constant Head

- percentage of any oversize material not used
- coefficient of permeability calculations
- laboratory form for test data (see Fig 3)
- test curves plotting velocity Q/At vs h/L
- corrected coefficients of permeability

Permeability - Falling Head

- description of apparatus
- all flow-rate measurements
- calculation of hydraulic conductivity

12. Data Requirements

Parameter	Detection Limit	Precision Desired (±% or Concentration)
Atterberg limits: liquid	Not Applicable	
plastic	Not Applicable	
grain size % finer or passing	1%	
consolidation	Not Applicable	
permeability (constant head)	Not Applicable	
permeability (falling head)	Not Applicable	

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Atterberg: lab duplicate	1 per 10 or less samples	20%
grain size: lab duplicate	1 per 10 or less samples	SIEVE ANALYSIS: *
consolidation: none	—	each coarse fraction 35% RPD
triaxial shear: none	—	each fine fraction 20% RPD
permeability: none	—	HYDROMETER ANALYSIS:
		each fraction: 10% RPD

* coarse fraction: > No. 10
fine fraction: < No. 10

14. Action Required if Limits are Exceeded

Reanalyze; Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

PARTICLE SIZE

SAMPLE PREPARATION

ATTACHMENT 1

- 1.) Air-dry entire sample (see below), record air-dried weight (A)
- 2.) If the sample contains large pieces of material other than soil such as wood, leaves, leather, mats of hair, etc., this material should be removed by the analyst after the original air-dried weight has been recorded. A short written description of the material removed should be recorded. Record weight of material removed (B).

3.) Record weight of air-dried soil submitted, (A) - (B).

NOTE: Air-dry sample by spreading out sample in an aluminum pan or a crucible to dry. Length of time to air-dry sample (24 hours to possibly several days) will depend on type of soil received and its moisture content. Final air-dried soil should have <5% moisture.

MINIMUM SAMPLE SIZES

The air-dried soil is prepared for mechanical analysis as described in ASTM D-421. The separation on the No. 10 sieve results in a coarse and a fine fraction.

ASTM D-422 gives minimum sample sizes for samples with nominal diameters of the largest particles ranging from 3/8" to 3". Furthermore, these samples as well as samples with the largest particles less than 3/8" require a minimum sample size to yield portions passing a No. 10 sieve of 115 grams for sandy soils and 65 grams for silt/clay soils.

It is acceptable to use entire sample for analysis to meet these minimum requirements.

If these minimum requirements are not possible due to insufficient sample size, OR if the total weight of air-dried soil submitted is less than 200 grams, CONTACT SMO IMMEDIATELY (SMO will contact the Region). Give SMO the following information for the samples not meeting the ASTM D-422 requirements or the 200 gram requirement: sample numbers, sample weights, and required sample weights. Region V CRL will determine whether to proceed with or terminate analysis.

COARSE FRACTION:

Perform a sieve analysis of the portion retained on the No. 10 sieve according to the ASTM test method using the following sieves: 3" (75 mm), 2" (50 mm), 1 1/2" (37.5 mm), 1" (25.0 mm), 3/4" (19.0 mm), 3/8" (9.5 mm), No. 4 (4.75 mm), No. 10 (2.00 mm).

FINE FRACTION:

Perform hydrometer, hygroscopic moisture, and specific gravity analysis on the fraction that passes a No. 10 sieve. Give a written description of the portion passing No. 10 sieve. If sandy, use 100 grams for hydrometer analysis, and if clay/silt, use 50 grams for hydrometer analysis.

Perform a sieve analysis of the portion that passes a No. 10 sieve according to ASTM D-422 using the following sieves: No. 20, No. 40, No. 60, No. 80, No. 100, No. 200.

PARTICLE SIZE

ATTACHMENT 2

RAW DATA:

All hand-written raw data must be submitted, and should include the following:

- 1.) Total air-dried weight of sample submitted (A).
- 2.) Total air-dried weight of extraneous material removed (B).
- 3.) Total air-dried weight of soil submitted (A) - (B).
- 4.) Total weight of soil retained on a No. 10 sieve.
- 5.) Total weight of soil passing a No. 10 sieve.
- 6.) Complete coarse fraction sieve analysis.
- 7.) Complete hydrometer analysis including time, temperature, hydrometer readings, type of hydrometer, etc.
- 8.) Complete hygroscopic moisture analysis.
- 9.) Complete specific gravity analysis.
- 10.) Complete fine fraction sieve analysis.
- 11.) Written description of extraneous material removed (if any), and of material passing a No. 10 sieve (sand or clay/silt).

Attachment 3 is suggested to be used as a raw data form by the laboratory, or the laboratory may use their own raw data forms as long as all required deliverables are included.

CALCULATIONS:

All calculations may be performed using a computer generated spreadsheet, but both handwritten raw data AND computer spreadsheets must be submitted as case deliverables.

REPORT:

Report results as percent finer than the specified particle size. Present data as tabulated AND in the form of a grain-sized distribution curve on a semi-logarithmic chart with percent finer by weight plotted on the arithmetic scale and grain size plotted on the logarithmic scale.

ASTM D-422 Particle Size Analysis of Soils

 Laboratory: _____
 Analyst: _____

EPA Sample No.: _____

Lab Sample No.: _____

Sample Preparation:

Total air-dried sample:	_____	g
Total air-dried extraneous material:	_____	g
Total air-dried soil:	_____	g
Total weight soil retained No. 10 sieve:	_____	g
Total weight soil passing No. 10 sieve:	_____	g
% Retained No. 10 Sieve:	_____	%
% Passing No. 10 Sieve:	_____	%

 Extraneous material description:
 % passing No. 10 description : _____

Coarse Sieve Analysis:

Date: _____

Sieve Size

Weight Retained, g

 3"
 2"
 1 1/2"
 1"
 3/4"
 3/8"
 No. 4
 No. 10

Hydrometer Analysis: Date: _____ Type of hydrometer: _____

Time (min.)	Actual Time	Hydrometer Actual	Hydrometer Comp. Corr.	Corrected Hydrometer	Temperature °C
2	_____	_____	_____	_____	_____
5	_____	_____	_____	_____	_____
15	_____	_____	_____	_____	_____
30	_____	_____	_____	_____	_____
60	_____	_____	_____	_____	_____
250	_____	_____	_____	_____	_____
1440	_____	_____	_____	_____	_____

Fine Sieve Analysis:

Date: _____

Sieve Size:

Weight Retained, g

 No. 20
 No. 40
 No. 60
 No. 80
 No. 100
 No. 200

Hygroscopic Moisture Analysis:

Tare:	_____	g
Sample Weight:	_____	g
Tare + Dry Weight:	_____	g
Dry Weight:	_____	g
% Moisture:	_____	%

Specific Gravity Analysis:

Pycnometer:	_____	g
Pycnometer, Water:	_____	g
Pycnometer, Water, Soil:	_____	g
Temperature, °C:	_____	
Specific Gravity:	_____	

Comments: _____



Standard Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants¹

This standard is issued under the fixed designation D 421; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the dry preparation of soil samples as received from the field for particle-size analysis and the determination of the soil constants.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards

D 2217 Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

3. Significance and Use

3.1 This practice can be used to prepare samples for particle-size and plasticity tests where it is desired to determine test values on air-dried samples, or where it is known that air drying does not have an effect on test results relative to samples prepared in accordance with Practice D 2217.

4. Apparatus

4.1 *Balance*, sensitive to 0.1 g.

4.2 *Mortar and Rubber-Covered Pestle*, suitable for breaking up the aggregations of soil particles.

4.3 *Sieves*—A series of sieves, of square mesh woven wire cloth, conforming to Specification E 11. The sieves required are as follows:

No. 4 (4.75-mm)
No. 10 (2.00-mm)
No. 40 (425- μ m)

4.4 *Sampler*—A riffle sampler or sample splitter, for quartering the samples.

5. Sampling

5.1 Expose the soil sample as received from the field to the air at room temperature until dried thoroughly. Break up the aggregations thoroughly in the mortar with a rubber-covered pestle. Select a representative sample of the amount required to perform the desired tests by the method of quartering or by the use of a sampler. The amounts of material required to perform the individual tests are as follows:

5.1.1 *Particle-Size Analysis*—For the particle-size analysis, material passing a No. 10 (2.00-mm) sieve is required in amounts equal to 115 g of sandy soils and 65 g of either silt or clay soils.

5.1.2 *Tests for Soil Constants*—For the tests for soil constants, material passing the No. 40 (425- μ m) sieve is required in total amount of 220 g, allocated as follows:

Test	Grams
Liquid limit	100
Plastic limit	15
Centrifuge moisture equivalent	10
Volumetric shrinkage	30
Check tests	65

6. Preparation of Test Sample

6.1 Select that portion of the air-dried sample selected for purpose of tests and record the mass as the mass of the total test sample uncorrected for hygroscopic moisture. Separate the test sample by sieving with a No. 10 (2.00-mm) sieve. Grind that fraction retained on the No. 10 sieve in a mortar with a rubber-covered pestle until the aggregations of soil particles are broken up into the separate grains. Then separate the ground soil into two fractions by sieving with a No. 10 sieve.

6.2 Wash that fraction retained after the second sieving free of all fine material, dry, and weigh. Record this mass as the mass of coarse material. Sieve the coarse material, after being washed and dried, on the No. 4 (4.75-mm) sieve and record the mass retained on the No. 4 sieve.

7. Test Sample for Particle-Size Analysis

7.1 Thoroughly mix together the fractions passing the No. 10 (2.00-mm) sieve in both sieving operations, and by the method of quartering or the use of a sampler, select a portion weighing approximately 115 g for sandy soils and approximately 65 g for silt and clay soil for particle-size analysis.

8. Test Sample for Soil Constants

8.1 Separate the remaining portion of the material passing the No. 10 (2.00-mm) sieve into two parts by means of a No. 40 (425- μ m) sieve. Discard the fraction retained on the No.

¹ This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

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² Annual Book of ASTM Standards, Vol 04.08

³ Annual Book of ASTM Standards, Vol 14.02

40 sieve. Use the fraction passing the No. 40 sieve for the determination of the soil constants.

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Standard Method for Particle-Size Analysis of Soils¹

This standard is issued under the fixed designation D 422; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Section 2 was added editorially and subsequent sections renumbered in July 1984.

1. Scope

1.1 This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Notes 1 and 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425- μm), or No. 200 (75- μm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (1) a high-speed mechanical stirrer, and (2) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20- μm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending on soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 μm .

2. Referenced Documents

2.1 ASTM Standards²

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants³

E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

E 100 Specification for ASTM Hydrometers⁴

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a

replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than $\frac{1}{4}$ in. (19.0 mm) nor more than $1\frac{1}{2}$ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup⁵ (Note 3) conforming to the general details shown in Fig. 3 (Notes 4 and 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft³/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E 11. A full set of sieves includes the following (Note 6):

¹ This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

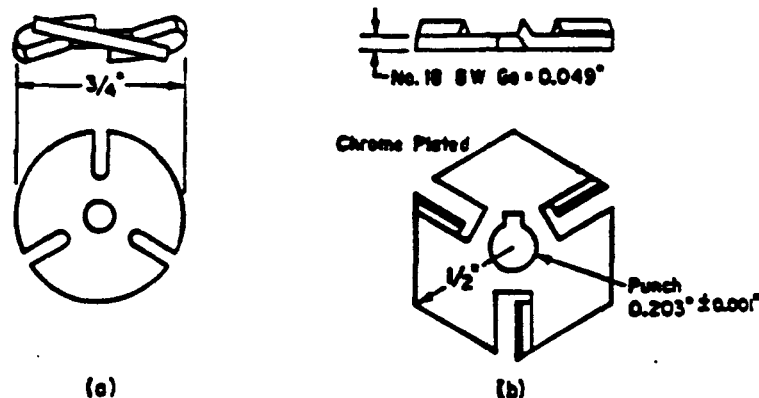
Current edition approved Nov. 21, 1963. Originally published 1935. Replaces - 62.

² Annual Book of ASTM Standards, Vol 04.08

³ Annual Book of ASTM Standards, Vol 14.02

⁴ Annual Book of ASTM Standards, Vol 14.01.

⁵ Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103. Order Adjunct No. 12-404220-00.



Metric Equivalents					
in.	0.001	0.049	0.203	1/2	3/4
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles

3-in. (75-mm)	No. 10 (2.00-mm)
2-in. (50-mm)	No. 20 (850-μm)
1 1/2-in. (37.5-mm)	No. 40 (425-μm)
1-in. (25.0-mm)	No. 60 (250-μm)
3/4-in. (19.0-mm)	No. 140 (106-μm)
1/2-in. (9.5-mm)	No. 200 (75-μm)
No. 4 (4.75-mm)	

NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)	No. 16 (1.18-mm)
1 1/2-in. (37.5-mm)	No. 30 (600-μm)
1-in. (25.0-mm)	No. 50 (300-μm)
3/4-in. (19.0-mm)	No. 100 (150-μm)
1/2-in. (9.5-mm)	No. 200 (75-μm)
No. 4 (4.75-mm)	
No. 8 (2.36-mm)	

3.7 Water Bath or Constant-Temperature Room—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 Beaker—A beaker of 250-mL capacity.

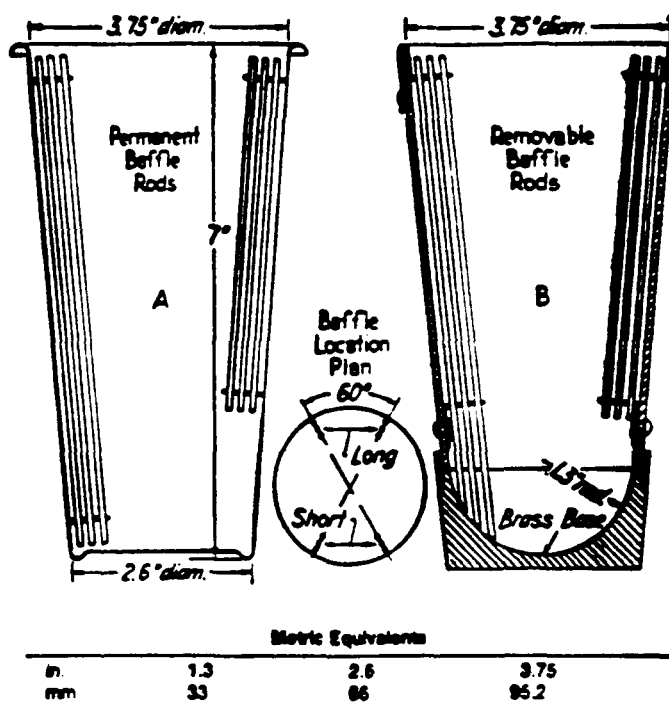
3.9 Timing Device—A watch or clock with a second hand.

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall



Metric Equivalents			
in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

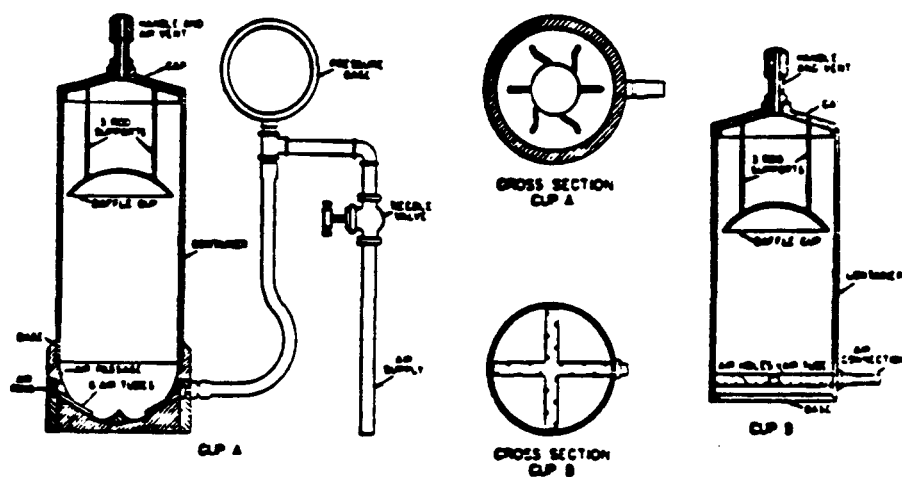


FIG. 3 Air-Jet Dispersion Cups of Apparatus B

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D 421. During the preparation procedure the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Practice D 421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
3/4 (19.0)	500
1/2 (12.5)	1000
1 (25.4)	2000
1 1/2 (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

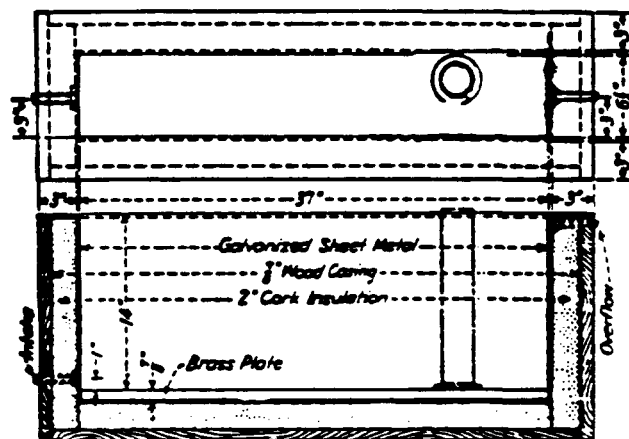
5.2 Provision is made in Section 5 of Practice D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

6. Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm),



Metric Equivalents						
in.	1/4	1	3	6 1/4	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath

2-in. (50.8-mm), 1 1/2-in. (37.5-mm), 1-in. (25.4-mm), 3/4-in. (19.0-mm), 1/2-in. (12.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-mm) SIEVE

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/L). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil - water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil - water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil - water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil - water slurry to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil - water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns.

Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75- μ m) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the $\frac{1}{2}$ -in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the $\frac{1}{2}$ -in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

12.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

13. Hygroscopic Moisture Correction Factor

13.1 The hygroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

14. Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

TABLE 1 Values of Correction Factor, a , for Different Specific Gravities of Soil Particles^a

Specific Gravity	Correction Factor ^a
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

^a For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = [(100\,000/W) \times G/(G - G_1)](R - G_1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W) \times 100$$

where:

a = correction fraction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1).

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension.

R = hydrometer reading with composite correction applied (Section 7).

W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g.

G = specific gravity of the soil particles, and

G_1 = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_1 .

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. According to Stokes' law:

$$D = \sqrt{[30\eta/980(G - G_1)] \times L/T}$$

where:

- D = diameter of particle, mm,
 n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),
 L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (Table 2)).
 T = interval of time from beginning of sedimentation to the taking of the reading, min.
 G = specific gravity of soil particles, and
 G_s = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

15.2 For convenience in calculations the above equation may be written as follows:

$$D = K\sqrt{L/T}$$

where:

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A - and B -scales, the square root being indicated on the D -scale. Without ascertaining the value of the square root it may be multiplied by K , using either the C - or CI -scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes^a

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

^a Values of effective depth are calculated from the equation:

$$L = L_1 + \frac{1}{2}(L_2 - (V_0/A))$$

where:

- L = effective depth, cm.
 L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm.
 L_2 = overall length of the hydrometer bulb, cm.
 V_0 = volume of hydrometer bulb, cm³, and
 A = cross-sectional area of sedimentation cylinder, cm²

Values used in calculating the values in Table 2 are as follows:

For both hydrometers, 151H and 152H:

L_2 = 14.0 cm

V_0 = 67.0 cm³

A = 27.8 cm²

For hydrometer 151H:

L_1 = 10.5 cm for a reading of 1.000

= 2.3 cm for a reading of 1.031

For hydrometer 152H:

L_1 = 10.5 cm for a reading of 0 g/litre

= 2.3 cm for a reading of 50 g/litre

17. Graph

17.1 When the hydrometer analysis is performed, a graph

TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

Temperature, °C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01384	0.01374	0.01366
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01366	0.01358
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.01323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01308	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01225	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01255	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles.

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16).

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular.

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable.

18.1.4 Specific gravity, if unusually high or low.

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed

almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

- | | |
|--------------------------------------------------------------------|---------|
| (1) Gravel, passing 3-in. and retained on No. 4 sieve | % |
| (2) Sand, passing No. 4 sieve and retained on No. 200 sieve | % |
| (a) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve | % |
| (b) Medium sand, passing No. 10 sieve and retained on No. 40 sieve | % |
| (c) Fine sand, passing No. 40 sieve and retained on No. 200 sieve | % |
| (3) Silt size, 0.074 to 0.005 mm | % |
| (4) Clay size, smaller than 0.005 mm | % |
| Colloids, smaller than 0.001 mm | % |

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.
2-in.
1½-in.
1-in.
¾-in.
½-in.
No. 4 (4.75-mm)
No. 10 (2.00-mm)
No. 40 (425-µm)
No. 200 (75-µm)

HYDROMETER ANALYSIS

0.074 mm
0.005 mm
0.001 mm

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-µm) sieves may be substituted for No. 10 and No. 40 sieves.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Specific Gravity of Soils¹

This standard is issued under the fixed designation D 854; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers determination of the specific gravity of soils by means of a pycnometer. When the soil is composed of particles larger than the No. 4 (4.75-mm) sieve, the method outlined in Test Method C 127 shall be followed. When the soil is composed of particles both larger and smaller than the No. 4 sieve, the sample shall be separated on the No. 4 sieve and the appropriate test method used on each portion. The specific gravity value for the soil shall be the weighted average of the two values (Note 1). When the specific gravity value is to be used in calculations in connection with the hydrometer portion of Method D 422, it is intended that the specific gravity test be made on that portion of the soil which passes the No. 10 (2.00-mm) sieve.

NOTE 1—The weighted average specific gravity should be calculated using the following equation:

$$G_{w_1} = \frac{1}{\frac{R_1}{100G_1} + \frac{P_1}{100G_2}}$$

where:

G_{w_1} = weighted average specific gravity of soils composed of particles larger and smaller than the No. 4 (4.75-mm) sieve,

R_1 = percent of soil particles retained on the No. 4 sieve,

P_1 = percent of soil particles passing the No. 4 sieve,

G_1 = apparent specific gravity of soil particles retained on the No. 4 sieve as determined by Test Method C 127, and

G_2 = specific gravity of soil particles passing the No. 4 sieve as determined by this test method.

1.2 The values stated in acceptable metric units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

¹ This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved Nov. 28, 1983. Published January 1984. Originally issued as D 854 - 45. Last previous edition D 854 - 58 (1979).

2. Referenced Documents

2.1 ASTM Standards:

C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate²

C 670 Practice for Preparing Precision Statements for Test Methods for Construction Materials²

D 422 Method for Particle-Size Analysis of Soils³

E 12 Definitions of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases⁴

3. Definition

3.1 *specific gravity*—the ratio of the mass of a unit volume of a material at a stated temperature to the mass in air of the same volume of gas-free distilled water at a stated temperature (per Definitions E 12).

4. Significance and Use

4.1 The specific gravity of a soil is used in almost every equation expressing the phase relationship of air, water, and solids in a given volume of material.

4.2 The term "solid particles," as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not very soluble in water. Therefore, the specific gravity of materials containing extraneous matter (such as cement, lime, etc.), water-soluble matter (such as sodium chloride), and soils containing matter with a specific gravity of less than one, typically require special treatment or a qualified definition of specific gravity.

5. Apparatus

5.1 *Pycnometer*—Either a volumetric flask having a capacity of at least 100 mL or a stoppered bottle having a capacity of at least 50 mL (Note 2). The stopper shall be of the same material as the bottle, and of such size and shape that it can be easily inserted to a fixed depth in the neck of the bottle, and shall have a small hole through its center to permit the emission of air and surplus water.

NOTE 2—The use of either the volumetric flask or the stoppered bottle is a matter of individual preference, but in general, the flask should be used when a larger sample than can be used in the stoppered bottle is needed due to maximum grain size of the sample.

² Annual Book of ASTM Standards, Vol 04.02

³ Annual Book of ASTM Standards, Vol 04.08

⁴ Annual Book of ASTM Standards, Vol 15.05

5.2 **Balance**—Either a balance sensitive to 0.01 g for use with the volumetric flask, or a balance sensitive to 0.001 g for use with the stoppered bottle.

6. Calibration of Pycnometer

6.1 The pycnometer shall be cleaned, dried, weighed, and the weight recorded. The pycnometer shall be filled with distilled water (Note 3) essentially at room temperature. The weight of the pycnometer and water, W_2 , shall be determined and recorded. A thermometer shall be inserted in the water and its temperature T , determined to the nearest whole degree.

NOTE 3—Kerosine is a better wetting agent than water for most soils and may be used in place of distilled water for oven-dried samples.

6.2 From the weight W_2 determined at the observed temperature T , a table of values of weights W_2 shall be prepared for a series of temperatures that are likely to prevail when weights W_1 are determined later (Note 4). These values of W_2 shall be calculated as follows:

$$W_2 \text{ (at } T_2) = (\text{density of water at } T_2 / \text{density of water at } T_1) \times (W_2 \text{ (at } T_1) - W_1) + W_1$$

where:

W_2 = weight of pycnometer and water, g.

W_1 = weight of pycnometer, g.

T_1 = observed temperature of water, °C, and

T_2 = any other desired temperature, °C.

NOTE 4—This method provides a procedure that is most convenient for laboratories making many determinations with the same pycnometer. It is equally applicable to a single determination. Bringing the pycnometer and contents to some designated temperature when weights W_1 and W_2 are taken, requires considerable time. It is much more convenient to prepare a table of weights W_2 for various temperatures likely to prevail when weights W_1 are taken. It is important that weights W_1 and W_2 be based on water at the same temperature. Values for the relative density of water at temperatures from 18 to 30°C are given in Table 1.

7. Sampling

7.1 The soil to be used in specific gravity test may contain its natural moisture or be oven-dried. The weight of the test sample on an oven-dry basis shall be at least 25 g when the volumetric flask is to be used, and at least 10 g when the stoppered bottle is to be used.

7.2 **Samples Containing Natural Moisture**—When the sample contains its natural moisture, the weight of the soil,

W_s , on an oven-dry basis shall be determined at the end of the test by evaporating the water in an oven maintained at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) (Note 5). Samples of clay soils containing their natural moisture content shall be dispersed in distilled water before placing in the flask, using the dispersing equipment specified in Method D 422 (Note 6).

7.3 **Oven-Dried Samples**—When an oven-dried sample is to be used, the sample shall be dried for at least 12 h, or to constant weight, in an oven maintained at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) (Note 5), cooled in a desiccator, and weighed upon removal from the desiccator. The sample shall then be soaked in distilled water for at least 12 h.

NOTE 5—Drying of certain soils at 110°C may bring about loss of moisture of composition or hydration, and in such cases drying shall be done, if desired, in reduced air pressure and at a lower temperature.

NOTE 6—The minimum volume of slurry that can be prepared by the dispersing equipment specified in Method D 422 is such that a 500-mL flask is needed as the pycnometer.

8. Procedure

8.1 Place the sample in the pycnometer, taking care not to lose any of the soil in case the weight of the sample has been determined. Add distilled water to fill the volumetric flask about three-fourths full or the stoppered bottle about half full.

8.2 Remove entrapped air by either of the following methods: (1) subject the contents to a partial vacuum (air pressure not exceeding 100 mm Hg) or (2) boil gently for at least 10 min while occasionally rolling the pycnometer to assist in the removal of the air. Subject the contents to reduced air pressure either by connecting the pycnometer directly to an aspirator or vacuum pump, or by use of a bell jar. Some soils boil violently when subjected to reduced air pressure. It will be necessary in those cases to reduce the air pressure at a slower rate or to use a larger flask. Cool samples that are heated to room temperature.

8.3 Fill the pycnometer with distilled water, clean the outside and dry with a clean, dry cloth. Determine the weight of the pycnometer and contents, W_2 , and the temperature in degrees Celsius, T_2 , of the contents as described in Section 6.

9. Calculation and Report

9.1 Calculate the specific gravity of the soil, based on water at a temperature T_s , as follows:

$$\text{Specific gravity, } T_s/T_s = W_s / [W_2 + (W_1 - W_2)]$$

where:

W_s = weight of sample of oven-dry soil, g.

W_2 = weight of pycnometer filled with water at temperature T_2 (Note 7), g.

W_1 = weight of pycnometer filled with water and soil at temperature T_2 , g, and

T_s = temperature of the contents of the pycnometer when weight W_s was determined, °C.

NOTE 7—This value shall be taken from the table of values of W_2 prepared in accordance with 6.2, for the temperature prevailing when weight W_2 was taken.

9.2 Unless otherwise required, specific gravity values reported shall be based on water at 20°C . The value based on water at 20°C shall be calculated from the value based on water at the observed temperature T_s , as follows:

TABLE 1 Relative Density of Water and Conversion Factor K For Various Temperatures

Temperature, °C	Relative Density of Water	Conversion Factor K
18	0.9986244	1.0004
19	0.9984347	1.0002
20	0.9982343	1.0000
21	0.9980233	0.9998
22	0.9978019	0.9996
23	0.9975702	0.9993
24	0.9973286	0.9991
25	0.9970770	0.9989
26	0.9968156	0.9986
27	0.9965451	0.9983
28	0.9962652	0.9980
29	0.9959761	0.9977
30	0.9956780	0.9974

Specific gravity, $T_w/20^\circ\text{C} = K \times \text{specific gravity, } T_w/T_s$

where:

K = a number found by dividing the relative density of water at temperature T_s by the relative density of water at 20°C . Values for a range of temperatures are given in Table 1.

9.3 When it is desired to report the specific gravity value based on water at 4°C , such a specific gravity value may be calculated by multiplying the specific gravity value at temperature T_s by the relative density of water at temperature T_s .

9.4 When any portion of the original sample of soil is eliminated in the preparation of the test sample, the portion on which the test has been made shall be reported.

10. Precision and Bias

10.1 Criteria for judging the acceptability of specific gravity test results obtained by this test method on material passing

the No. 4 (4.75-mm) sieve are given as follows (Note 8):

Material and Type Index	Standard Deviation ^a	Acceptable Range of Two Results (percent of mass) ^b
<i>Single-operator precision:</i>		
Cohesive soils	0.021	0.06
Noncohesive soils	"	"
<i>Multilaboratory precision:</i>		
Cohesive soils	0.056	0.16
Noncohesive soils	"	"

^a These numbers represent, respectively, the (1S) and (2S) limits as described in Practice C 670.

^b Criteria for assigning standard deviation values for non-cohesive soils are not available at the present time.

NOTE 8—The figures given in Column 2 are the standard deviations that have been found to be appropriate for the materials described in Column 1. The figures given in Column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests.

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Designation: D 4318 - 84

Standard Test Method for LIQUID LIMIT, PLASTIC LIMIT, AND PLASTICITY INDEX OF SOILS¹

This standard is issued under the fixed designation D 4318; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscripted epsilon (ϵ) indicates an editorial change since the last revision or approval.

1. Scope

1.1 This test method covers the determination of the liquid limit, plastic limit, and the plasticity index of soils as defined in Section 3.

1.1.1 Two procedures for preparing test specimens and two procedures for performing the liquid limit are provided as follows:

- A Multipoint test using a wet preparation procedure, described in Sections 10.1, 11, and 12.
- B Multipoint test using a dry preparation procedure, described in Sections 10.2, 11, and 12.
- C One-point test using a wet preparation procedure, described in Sections 13, 14, and 15.
- D One-point test using a dry preparation procedure, described in Sections 13, 14, and 15.

The procedure to be used shall be specified by the requesting authority. If no procedure is specified, Procedure A shall be used.

NOTE 1—Prior to the adoption of this test method, a curved grooving tool was specified as part of the apparatus for performing the liquid limit test. The curved tool is not considered to be as accurate as the flat tool described in 6.2 since it does not control the depth of the soil in the liquid limit cup. However, there are some data which indicate that typically the liquid limit is slightly increased when the flat tool is used instead of the curved tool.

1.1.2 The plastic limit test procedure is described in Sections 16, 17, and 18. The plastic limit test is performed on material prepared for the liquid limit test. In effect, there are two procedures for preparing test specimens for the plastic limit test.

1.1.3 The procedure for calculating the plasticity index is given in Section 19.

1.2 The liquid limit and plastic limit of soils (along with the shrinkage limit) are often collectively referred to as the *Aterberg limits* in recognition of their formation by Swedish soil scientist, A. Aterberg. These limits distinguish the boundaries of the several consistency states of plastic soils.

1.3 As used in this test method, soil is any natural aggregation of mineral or organic materials, mixtures of such materials, or artificial mixtures of aggregates and natural mineral and organic particles.

1.4 The multipoint liquid limit procedure is somewhat more time consuming than the one-point procedure when both are performed by experienced operators. However, the one-point procedure requires the operator to judge when the test specimen is approximately at its liquid limit. In cases where this is not done reliably, the multipoint procedure is as fast as the one-point procedure and provides additional precision due to the information obtained from additional trials. It is particularly recommended that the multipoint procedure be used by inexperienced operators.

1.5 The correlations on which the calculations of the one-point procedure are based may not be valid for certain soils, such as organic soils or soils from a marine environment. The liquid

¹ This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rocks and is the direct responsibility of Subcommittee D18.03 on Test Methods, Procedures, and Devices Characterization of Soils.

Current edition approved Oct. 26, 1984. Published December 1984. Originally published as D 4318 - 83. Last previous edition D 4318 - 83⁷.

limit of these soils should therefore be determined by the multipoint procedure (Procedure A).

1.6 The liquid and plastic limits of many soils that have been allowed to dry before testing may be considerably different from values obtained on undried samples. If the liquid and plastic limits of soils are used to correlate or estimate the engineering behavior of soils in their natural moist state, samples should not be permitted to dry before testing unless data on dried samples are specifically desired.

1.7 The composition and concentration of soluble salts in a soil affect the values of the liquid and plastic limits as well as the water content values of soils (see Method D 2216). Special consideration should therefore be given to soils from a marine environment or other sources where high soluble salt concentrations may be present. The degree to which the salts present in these soils are diluted or concentrated must be given consideration if meaningful results are to be obtained.

1.8 Since the tests described herein are performed only on that portion of a soil which passes the 425- μ m (No. 40) sieve, the relative contribution of this portion of the soil to the properties of the sample as a whole must be considered when using these tests to evaluate the properties of a soil.

1.9 The values stated in acceptable metric units are to be regarded as the standard. The values given in parentheses are for information only.

1.10 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 ASTM Standards:

C 702 Methods for Reducing Field Samples of Aggregate to Testing Size¹

D 75 Practice for Sampling Aggregates²

D 420 Recommended Practice for Investigating and Sampling Soil and Rock for Engineering Purposes³

D 653 Terms and Symbols Relating to Soil and Rock Mechanics⁴

D 1241 Specification for Materials for Soil-Aggregate Subbase, Base, and Surface Courses⁵

D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures⁶

D 2240 Test Method for Rubber Property—Durometer Hardness⁷

D 2487 Test Method for Classification of Soils for Engineering Purposes⁸

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)⁹

D 3282 Practice for Classification of Soils and Soil-Aggregate Mixtures for Highway Construction Purposes¹⁰

E 11 Specification for Wire-Cloth Sieves for Testing Purposes¹¹

E 319 Methods of Testing Single-Arm Balances¹²

E 898 Method of Testing Top-Loading, Direct-Reading Laboratory Scales and Balances¹³

3. Definitions

3.1 *Atterberg limits*—originally, seven “limits of consistency” of fine-grained soils were defined by Albert Atterberg. In current engineering usage, the term usually refers only to the liquid limit, plastic limit, and in some references, the shrinkage limit.

3.2 *consistency*—the relative ease with which a soil can be deformed.

3.3 *liquid limit (LL)*—the water content, in percent, of a soil at the arbitrarily defined boundary between the liquid and plastic states. This water content is defined as the water content at which a pat of soil placed in a standard cup and cut by a groove of standard dimensions will flow together at the base of the groove for a distance of 13 mm (½ in.) when subjected to 25 shocks from the cup being dropped 10 mm in a standard liquid limit apparatus operated at a rate of 2 shocks per second.

¹ Annual Book of ASTM Standards, Vol 04.02.

² Annual Book of ASTM Standards, Vols 04.02, 04.03, and 04.08.

³ Annual Book of ASTM Standards, Vol 04.08.

⁴ Annual Book of ASTM Standards, Vol 09.01.

⁵ Annual Book of ASTM Standards, Vol 14.02.



NOTE 2—The undrained shear strength of soil at the liquid limit is considered to be 2 ± 0.2 kPa (0.28 psi).

3.4 *plastic limit (PL)*—the water content, in percent, of a soil at the boundary between the plastic and brittle states. The water content at this boundary is the water content at which a soil can no longer be deformed by rolling into 3.2 mm (1/8 in.) in diameter threads without crumbling.

3.5 *plastic soil*—a soil which has a range of water content over which it exhibits plasticity and which will retain its shape on drying.

3.6 *plasticity index (PI)*—the range of water content over which a soil behaves plastically. Numerically, it is the difference between the liquid limit and the plastic limit.

3.7 *liquid limit index*—the ratio, expressed as a percentage, of (1) the natural water content of a soil minus its plastic limit, to (2) its plasticity index.

3.8 *activity number (A)*—the ratio of (1) the plasticity index of a soil to (2) the percent by weight of particles having an equivalent diameter smaller than 0.002 mm.

4. Summary of Method

4.1 The sample is processed to remove any material retained on a 425- μ m (No. 40) sieve. The liquid limit is determined by performing trials in which a portion of the sample is spread in a brass cup, divided in two by a grooving tool, and then allowed to flow together from the shocks caused by repeatedly dropping the cup in a standard mechanical device. The multipoint liquid limit, Procedures A and B, requires three or more trials over a range of water contents to be performed and the data from the trials plotted or calculated to make a relationship from which the liquid limit is determined. The one-point liquid limit, Procedures C and D, uses the data from two trials at one water content multiplied by a correction factor to determine the liquid limit.

4.2 The plastic limit is determined by alternately pressing together and rolling into a 3.2 mm (1/8 in.) diameter thread a small portion of plastic soil until its water content is reduced to a point at which the thread crumbles and is no longer able to be pressed together and rerolled. The water content of the soil at this stage is reported as the plastic limit.

4.3 The plasticity index is calculated as the difference between the liquid limit and the plastic limit.

5. Significance and Use

5.1 This test method is used as an integral part of several engineering classification systems to characterize the fine-grained fractions of soils (see Test Method D 2487 and Practice D 3282) and to specify the fine-grained fractions of construction materials (see Specification D 1241). The liquid limit, plastic limit, and plasticity index of soils are also used extensively, either individually or together with other soil properties to correlate with engineering behavior such as compressibility, permeability, compactibility, shrink-swell, and shear strength.

5.2 The liquid and plastic limits of a soil can be used with the natural water content of the soil to express its relative consistency or liquidity index and can be used with the percentage finer than 2- μ m size to determine its activity number.

5.3 The one-point liquid limit procedure is frequently used for routine classification purposes. When greater precision is required, as when used for the acceptance of a material or for correlation with other test data, the multipoint procedure should be used.

5.4 These methods are sometimes used to evaluate the weathering characteristics of clay-shale materials. When subjected to repeated wetting and drying cycles, the liquid limits of these materials tend to increase. The amount of increase is considered to be a measure of a shale's susceptibility to weathering.

5.5 The liquid limit of a soil containing substantial amounts of organic matter decreases dramatically when the soil is oven-dried before testing. Comparison of the liquid limit of a sample before and after oven-drying can therefore be used as a qualitative measure of organic matter content of a soil.

6. Apparatus

6.1 *Liquid Limit Device*—A mechanical device consisting of a brass cup suspended from a carriage designed to control its drop onto a hard rubber base. A drawing showing the essential features of the device and the critical dimensions is given in Fig. 1. The design of the device may vary provided that the essential functions are



preserved. The device may be operated either by a hand crank or by an electric motor.

6.1.1 Base—The base shall be hard rubber having a D Durometer hardness of 80 to 90, and a resilience such that an 8-mm ($\frac{5}{16}$ -in.) diameter polished steel ball, when dropped from a height of 25 cm (9.84 in.) will have an average rebound of at least 80 % but no more than 90 %. The test shall be conducted on the finished base with feet attached.

6.1.2 Feet—The base shall be supported by rubber feet designed to provide isolation of the base from the work surface and having an A Durometer hardness no greater than 60 as measured on the finished feet attached to the base.

6.1.3 Cup—The cup shall be brass and have a weight including cup hanger, of 185 to 215 g.

6.1.4 Cam—The cam shall raise the cup smoothly and continuously to its maximum height over a distance of at least 180° of cam rotation. The preferred cam motion is a uniformly accelerated lift curve. The design of the cam and follower combination shall be such that there is no upward or downward velocity of the cup when the cam follower leaves the cam.

NOTE 3—The cam and follower design in Fig. 1 is for uniform accelerated (parabolic) motion after contact and assures that the cup has no velocity at drop off. Other cam designs also provide this feature and may be used. However, if the cam-follower lift pattern is not known, zero velocity at drop off can be assured by carefully filing or machining the cam and follower so that the cup height remains constant over the last 30 to 45° of cam rotation.

6.1.5 Carriage—The cup carriage shall be constructed in a way that allows convenient but secure adjustment of the height of drop of the cup to 10 mm (0.394 in.). The cup hanger shall be attached to the carriage by means of a pin which allows removal of the cup and cup hanger for cleaning and inspection.

6.1.6 Optional Motor Drive—As an alternative to the hand crank shown in Fig. 1, the device may be equipped with a motor to turn the cam. Such a motor must turn the cam at 2 ± 0.1 revolutions per second, and must be isolated from the rest of the device by rubber mounts or in some other way that prevents vibration from the motor being transmitted to the rest of the apparatus. It must be equipped with an ON-OFF switch and a means of conveniently positioning the cam for height of drop adjustment. The results obtained using a motor-driven device

must not differ from those obtained using a manually operated device.

6.2 Flat Grooving Tool—A grooving tool having dimensions shown in Fig. 2. The tool shall be made of plastic or noncorroding metal. The design of the tool may vary as long as the essential dimensions are maintained. The tool may, but need not, incorporate the gage for adjusting the height of drop of the liquid limit device.

6.3 Gage—A metal gage block for adjusting the height of drop of the cup, having the dimensions shown in Fig. 3. The design of the tool may vary provided the gage will rest securely on the base without being susceptible to rocking, and the edge which contacts the cup during adjustment is straight, at least 10 mm ($\frac{3}{8}$ in.) wide, and without bevel or radius.

6.4 Containers—Small corrosion-resistant containers with snug-fitting lids for water content specimens. Aluminum or stainless steel cans 2.5 cm (1 in.) high by 5 cm (2 in.) in diameter are appropriate.

6.5 Balance—A balance readable to at least 0.01 g and having an accuracy of 0.03 g within three standard deviations within the range of use. Within any 15-g range, a difference between readings shall be accurate within 0.01 g (Notes 4 and 5).

NOTE 4—See Methods E 198 and E 319 for an explanation of terms relating to balance performance.

NOTE 5—For frequent use, a top-loading type balance with automatic load indication, readable to 0.01 g, and having an index of precision (standard deviation) of 0.003 or better is most suitable for this method. However, nonautomatic indicating equal-arm analytical balances and some small equal arm top pan balances having readability and reproducibility of 0.002 g or better provide the required accuracy when used with a weight set of ASTM Class 4 (National Bureau of Standards Class P) or better. Ordinary commercial and classroom type balances such as beam balances are not suitable for this method.

6.6 Storage Container—A container in which to store the prepared soil specimen that will not contaminate the specimen in any way, and which prevents moisture loss. A porcelain, glass, or plastic dish about 11.4 cm (4½ in.) in diameter and a plastic bag large enough to enclose the dish and be folded over is adequate.

6.7 Ground Glass Plate—A ground glass plate at least 30 cm (12 in.) square by 1 cm ($\frac{3}{8}$ in.) thick for mixing soil and rolling plastic limit threads.

6.8 Spatula—A spatula or pill knife having a

blade about 2 cm (¾ in.) wide by about 10 cm (4 in.) long. In addition, a spatula having a blade about 2.5 cm (1 in.) wide and 15 cm (6 in.) long has been found useful for initial mixing of samples.

6.9 *Sieve*—A 20.3 cm (8 in.) diameter, 425- μ m (No. 40) sieve conforming to the requirements of Specification E 11 and having a rim at least 5 cm (2 in.) above the mesh. A 2-mm (No. 10) sieve meeting the same requirements may also be needed.

6.10 *Wash Bottle*, or similar container for adding controlled amounts of water to soil and washing fines from coarse particles.

6.11 *Drying Oven*—A thermostatically controlled oven, preferably of the forced-draft type, capable of continuously maintaining a temperature of $110 \pm 5^\circ\text{C}$ throughout the drying chamber. The oven shall be equipped with a thermometer of suitable range and accuracy for monitoring oven temperature.

6.12 *Washing Pan*—A round, flat-bottomed pan at least 7.6 cm (3 in.) deep, slightly larger at the bottom than a 20.3-cm (8-in.) diameter sieve.

6.13 *Rod* (optional)—A metal or plastic rod or tube 3.2 mm (¼ in.) in diameter and about 10 cm (4 in.) long for judging the size of plastic limit threads.

7. Materials

7.1 A supply of distilled or demineralized water.

8. Sampling

8.1 Samples may be taken from any location that satisfies testing needs. However, Methods C 702, and Practice D 75, and Recommended Practice D 420 should be used as guides for selecting and preserving samples from various types of sampling operations. Samples which will be prepared using the wet preparation procedure, 10.1, must be kept at their natural water content prior to preparation.

8.2 Where sampling operations have preserved the natural stratification of a sample, the various strata must be kept separated and tests performed on the particular stratum of interest with as little contamination as possible from other strata. Where a mixture of materials will be used in construction, combine the various components in such proportions that the resultant sample represents the actual construction case.

8.3 Where data from this test method are to be used for correlation with other laboratory or field test data, use the same material as used for these tests where possible.

8.4 Obtain a representative portion from the total sample sufficient to provide 150 to 200 g of material passing the 425- μ m (No. 40) sieve. Free flowing samples may be reduced by the methods of quartering or splitting. Cohesive samples shall be mixed thoroughly in a pan with a spatula, or scoop and a representative portion scooped from the total mass by making one or more sweeps with a scoop through the mixed mass.

9. Calibration of Apparatus

9.1 Inspection of Wear:

9.1.1 *Liquid Limit Device*—Determine that the liquid limit device is clean and in good working order. The following specific points should be checked:

9.1.1.1 *Wear of Base*—The spot on the base where the cup makes contact should be worn no greater than 10 mm (¾ in.) in diameter. If the wear spot is greater than this, the base can be machined to remove the worn spot provided the resurfacing does not make the base thinner than specified in 6.1 and the other dimensional relationships are maintained.

9.1.1.2 *Wear of Cup*—The cup must be replaced when the grooving tool has worn a depression in the cup 0.1 mm (0.004 in.) deep or when the edge of the cup has been reduced to half its original thickness. Verify that the cup is firmly attached to the cup hanger.

9.1.1.3 *Wear of Cup Hanger*—Verify that the cup hanger pivot does not bind and is not worn to an extent that allows more than 3-mm (¼-in.) side-to-side movement of the lowest point on the rim.

9.1.1.4 *Wear of Cam*—The cam shall not be worn to an extent that the cup drops before the cup hanger (cam follower) loses contact with the cam.

9.1.2 *Grooving Tools*—Inspect grooving tools for wear on a frequent and regular basis. The rapidity of wear depends on the material from which the tool is made and the types of soils being tested. Sandy soils cause rapid wear of grooving tools; therefore, when testing these materials, tools should be inspected more frequently than for other soils. Any tool with a tip width greater than 2.1 mm must not be used. The depth

of the tip of the grooving tool must be 7.9 to 8.1 mm.

NOTE 6—The width of the tip of grooving tools is conveniently checked using a pocket-sized measuring magnifier equipped with a millimeter scale. Magnifiers of this type are available from most laboratory supply companies. The depth of the tip of grooving tools can be checked using the depth measuring feature of vernier calipers.

9.2 Adjustment of Height of Drop—Adjust the height of drop of the cup so that the point on the cup that comes in contact with the base rises to a height of 10 ± 0.2 mm. See Fig. 4 for proper location of the gage relative to the cup during adjustment.

NOTE 7—A convenient procedure for adjusting the height of drop is as follows: place a piece of masking tape across the outside bottom of the cup parallel with the axis of the cup hanger pivot. The edge of the tape away from the cup hanger should touch the spot on the cup that contacts the base. For new cups, placing a piece of carbon paper on the base and allowing the cup to drop several times will mark the contact spot. Attach the cup to the device and turn the crank until the cup is raised to its maximum height. Slide the height gage under the cup from the front, and observe whether the gage contacts the cup or the tape. See Fig. 4. If the tape and cup are both contacted, the height of drop is approximately correct. If not, adjust the cup until simultaneous contact is made. Check adjustment by turning the crank at 2 revolutions per second while holding the gage in position against the tape and cup. If a ringing or clicking sound is heard without the cup rising from the gage, the adjustment is correct. If no ringing is heard or if the cup rises from the gage, readjust the height of drop. If the cup rocks on the gage during this checking operation, the cam follower pivot is excessively worn and the worn parts should be replaced. Always remove tape after completion of adjustment operation.

MULTIPOINT LIQUID LIMIT—PROCEDURES A AND B

10. Preparation of Test Specimens

10.1 Wet Preparation—Except where the dry method of specimen preparation is specified (10.2), prepare specimens for test as described in the following sections.

10.1.1 Samples Passing the 425- μ m (No. 40) Sieve—When by visual and manual procedures it is determined that the sample has little or no material retained on a 425- μ m (No. 40) sieve, prepare a specimen of 150 to 200 g by mixing thoroughly with distilled or demineralized water on the glass plate using the spatula. If desired, soak soil in a storage dish with small amount of water to soften the soil before the start of mixing.

Adjust the water content of the soil to bring it to a consistency that would require 25 to 35 blows of the liquid limit device to close the groove (Note 8). If, during mixing, a small percentage of material is encountered that would be retained on a 425- μ m (No. 40) sieve, remove these particles by hand, if possible. If it is impractical to remove the coarser material by hand, remove small percentages (less than about 15 %) of coarser material by working the specimen through a 425- μ m (No. 40) sieve using a piece of rubber sheeting, rubber stopper, or other convenient device provided the operation does not distort the sieve or degrade material that would be retained if the washing method described in 10.1.2 were used. If larger percentages of coarse material are encountered during mixing, or it is considered impractical to remove the coarser material by the methods just described, wash the sample as described in 10.1.2. When the coarse particles found during mixing are concretions, shells, or other fragile particles, do not crush these particles to make them pass a 425- μ m (No. 40) sieve, but remove by hand or by washing. Place the mixed soil in the storage dish, cover to prevent loss of moisture, and allow to stand for at least 16 h (overnight). After the standing period and immediately before starting the test, thoroughly remix the soil.

NOTE 8—The time taken to adequately mix a soil will vary greatly, depending on the plasticity and initial water content. Initial mixing times of more than 30 min may be needed for stiff, fat clays.

10.1.2 Samples Containing Material Retained on a 425- μ m (No. 40) Sieve

10.1.2.1 Select a sufficient quantity of soil at natural water content to provide 150 to 200 g of material passing the 425- μ m (No. 40) sieve. Place in a pan or dish and add sufficient water to cover the soil. Allow to soak until all lumps have softened and the fines no longer adhere to the surfaces of the coarse particles (Note 9).

NOTE 9—In some cases, the cations of salts present in tap water will exchange with the natural cations in the soil and significantly alter the test results should tap water be used in the soaking and washing operations. Unless it is known that such cations are not present in the tap water, distilled or demineralized water should be used. As a general rule, water containing more than 100 mg/L of dissolved solids should not be used for washing operations.

10.1.2.2 When the sample contains a large percentage of material retained on the 425- μ m



(No. 40) sieve, perform the following washing operation in increments, washing no more than 0.5 kg (1 lb) of material at one time. Place the 425- μ m (No. 40) sieve in the bottom of the clean pan. Pour the soil water mixture onto the sieve. If gravel or coarse sand particles are present, rinse as many of these as possible with small quantities of water from a wash bottle, and discard. Alternatively, pour the soil water mixture over a 2-mm (No. 10) sieve nested atop the 425- μ m (No. 40) sieve, rinse the fine material through and remove the 2-mm (No. 10) sieve. After washing and removing as much of the coarser material as possible, add sufficient water to the pan to bring the level to about 13 mm (1/2 in.) above the surface of the 425- μ m (No. 40) sieve. Agitate the slurry by stirring with the fingers while raising and lowering the sieve in the pan and swirling the suspension so that fine material is washed from the coarser particles. Disaggregate fine soil lumps that have not slaked by gently rubbing them over the sieve with the fingertips. Complete the washing operation by raising the sieve above the water surface and rinsing the material retained with a small amount of clean water. Discard material retained on the 425- μ m (No. 40) sieve.

10.1.2.3 Reduce the water content of the material passing the 425- μ m (No. 40) sieve until it approaches the liquid limit. Reduction of water content may be accomplished by one or a combination of the following methods: (a) exposing the air currents at ordinary room temperature, (b) exposing to warm air currents from a source such as an electric hair dryer, (c) filtering in a Buckner funnel or using filter candles, (d) decanting clear water from surface of suspension, or (e) draining in a colander or plunger of paris dish lined with high retentivity, high wet-strength filter paper.¹ If a plunger of paris dish is used, take care that the dish never becomes sufficiently saturated that it fails to actively absorb water into its surface. Thoroughly dry dishes between uses. During evaporation and cooling, stir the sample often enough to prevent overdrying of the fringes and soil pinnacles on the surface of the mixture. For soil samples containing soluble salts, use a method of water reduction such as a or b that will not eliminate the soluble salts from the test specimen.

10.1.2.4 Thoroughly mix the material passing the 425- μ m (No. 40) sieve on the glass plate using the spatula. Adjust the water content of the mixture, if necessary, by adding small increments of

distilled or demineralized water or by allowing the mixture to dry at room temperature while mixing on the glass plate. The soil should be at a water content that will result in closure of the groove in 25 to 35 blows. Return the mixed soil to the mixing dish, cover to prevent loss of moisture, and allow to stand for at least 16 h. After the standing period, and immediately before starting the test, remix the soil thoroughly.

10.2 Dry Preparation:

10.2.1 Select sufficient soil to provide 150 to 200 g of material passing the 425- μ m (No. 40) sieve after processing. Dry the sample at room temperature or in an oven at a temperature not exceeding 60°C until the soil clods will pulverize readily. Disaggregation is expedited if the sample is not allowed to completely dry. However, the soil should have a dry appearance when pulverized. Pulverize the sample in a mortar with a rubber tipped pestal or in some other way that does not cause breakdown of individual grains. When the coarse particles found during pulverization are concretions, shells, or other fragile particles, do not crush these particles to make them pass a 425- μ m (No. 40) sieve, but remove by hand or other suitable means, such as washing.

10.2.2 Separate the sample on a 425- μ m (No. 40) sieve, shaking the sieve by hand to assure thorough separation of the finer fraction. Return the material retained on the 425- μ m (No. 40) sieve to the pulverizing apparatus and repeat the pulverizing and sieving operations as many times as necessary to assure that all finer material has been disaggregated and material retained on the 425- μ m (No. 40) sieve consists only of individual sand or gravel grains.

10.2.3 Place material remaining on the 425- μ m (No. 40) sieve after the final pulverizing operations in a dish and soak in a small amount of water. Stir the soil water mixture and pour over the 425- μ m (No. 40) sieve, catching the water and any suspended fines in the washing pan. Pour this suspension into a dish containing the dry soil previously sieved through the 425- μ m (No. 40) sieve. Discard material retained on the 425- μ m (No. 40) sieve.

10.2.4 Adjust the water content as necessary by drying as described in 10.1.2.3 or by mixing on the glass plate, using the spatula while adding increments of distilled or demineralized water.

¹ S and S 995 filter press, available in 32-cm circle, has proven satisfactory.



until the soil is at a water content that will result in closure of the groove in 25 to 35 blows.

10.2.5 Put soil in the storage dish, cover to prevent loss of moisture and allow to stand for at least 16 h. After the standing period, and immediately before starting the test, thoroughly remix the soil (Note 8).

11. Procedure

11.1 Place a portion of the prepared soil in the cup of the liquid limit device at the point where the cup rests on the base, squeeze it down, and spread it into the cup to a depth of about 10 mm at its deepest point, tapering to form an approximately horizontal surface. Take care to eliminate air bubbles from the soil pat but form the pat with as few strokes as possible. Heap the unused soil on the glass plate and cover with the inverted storage dish or a wet towel.

11.2 Form a groove in the soil pat by drawing the tool, beveled edge forward, through the soil on a line joining the highest point to the lowest point on the rim of the cup. When cutting the groove, hold the grooving tool against the surface of the cup and draw in an arc, maintaining the tool perpendicular to the surface of the cup throughout its movement. See Fig. 5. In soils where a groove cannot be made in one stroke without tearing the soil, cut the groove with several strokes of the grooving tool. Alternatively, cut the groove to slightly less than required dimensions with a spatula and use the grooving tool to bring the groove to final dimensions. Exercise extreme care to prevent sliding the soil pat relative to the surface of the cup.

11.3 Verify that no crumbs of soil are present on the base or the underside of the cup. Lift and drop the cup by turning the crank at a rate of 1.9 to 2.1 drops per second until the two halves of the soil pat come in contact at the bottom of the groove along a distance of 13 mm ($\frac{1}{2}$ in.). See Fig. 6.

NOTE 10—Use the end of the grooving tool, Fig. 2, or a scale to verify that the groove has closed 13 mm ($\frac{1}{2}$ in.).

11.4 Verify that an air bubble has not caused premature closing of the groove by observing that both sides of the groove have flowed together with approximately the same shape. If a bubble has caused premature closing of the groove, reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving

operation and repeat 11.1 to 11.3. If the soil slides on the surface of the cup, repeat 11.1 through 11.3 at a higher water content. If, after several trials at successively higher water contents, the soil pat continues to slide in the cup or if the number of blows required to close the groove is always less than 25, record that the liquid limit could not be determined, and report the soil as nonplastic without performing the plastic limit test.

11.5 Record the number of drops, N , required to close the groove. Remove a slice of soil approximately the width of the spatula, extending from edge to edge of the soil cake at right angles to the groove and including that portion of the groove in which the soil flowed together, place in a weighed container, and cover.

11.6 Return the soil remaining in the cup to the glass plate. Wash and dry the cup and grooving tool and reattach the cup to the carriage in preparation for the next trial.

11.7 Remix the entire soil specimen on the glass plate adding distilled water to increase the water content of the soil and decrease the number of blows required to close the groove. Repeat 11.1 through 11.6 for at least two additional trials producing successively lower numbers of blows to close the groove. One of the trials shall be for a closure requiring 25 to 35 blows, one for closure between 20 and 30 blows, and one trial for a closure requiring 15 to 25 blows.

11.8 Determine the water content, W , of the soil specimen from each trial in accordance with Method D 2216. Make all weighings on the same balance. Initial weighings should be performed immediately after completion of the test. If the test is to be interrupted for more than about 15 min, the specimens already obtained should be weighed at the time of the interruption.

12. Calculations

12.1 Plot the relationship between the water content, W , and the corresponding number of drops, N , of the cup on a semilogarithmic graph with the water content as ordinates on the arithmetical scale, and the number of drops as abscissas on the logarithmic scale. Draw the best straight line through the three or more plotted points.

12.2 Take the water content corresponding to the intersection of the line with the 25-drop abscissa as the liquid limit of the soil. Computa-



tional methods may be substituted for the graphical method for fitting a straight line to the data and determining the liquid limit.

ONE-POINT LIQUID LIMIT—PROCEDURES C AND D

13. Preparation of Test Specimens

13.1 Prepare the specimen in the same manner as described in Section 10, except that at mixing, adjust the water content to a consistency requiring 20 to 30 drops of the liquid limit cup to close the groove.

14. Procedure

14.1 Proceed as described in 11.1 through 11.5 except that the number of blows required to close the groove shall be 20 to 30. If less than 20 or more than 30 blows are required, adjust the water content of the soil and repeat the procedure.

14.2 Immediately after removing a water content specimen as described in 11.5, reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving and water content sampling operations. Repeat 11.2 through 11.5, and, if the second closing of the groove requires the same number of drops or no more than two drops difference, secure another water content specimen. Otherwise, remix the entire specimen and repeat.

NOTE 11—Excessive drying or inadequate mixing will cause the number of blows to vary.

14.3 Determine water contents of specimens as described in 11.8.

15. Calculations

15.1 Determine the liquid limit for each water content specimen using one of the following equations:

$$LL = W_p \left(\frac{N^*}{25} \right)^{0.121} \text{ or}$$

$$LL = K(W_p)$$

where:

N^* = the number of blows causing closure of the groove at water content.

W_p = water content, and

K = a factor given in Table 1.

The liquid limit is the average of the two trial liquid limit values.

15.2 If the difference between the two trial

liquid limit values is greater than one percentage point, repeat the test.

PLASTIC LIMIT

16. Preparation of Test Specimen

16.1 Select a 20-g portion of soil from the material prepared for the liquid limit test, either after the second mixing before the test, or from the soil remaining after completion of the test. Reduce the water content of the soil to a consistency at which it can be rolled without sucking to the hands by spreading and mixing continuously on the glass plate. The drying process may be accelerated by exposing the soil to the air current from an electric fan, or by blotting with paper that does not add any fiber to the soil, such as hard surface paper toweling or high wet strength filter paper.

17. Procedure

17.1 From the 20-g mass, select a portion of 1.5 to 2.0 g. Form the test specimen into an ellipsoidal mass. Roll this mass between the palm or fingers and the ground-glass plate with just sufficient pressure to roll the mass into a thread of uniform diameter throughout its length (Note 12). The thread shall be further deformed on each stroke so that its diameter is continuously reduced and its length extended until the diameter reaches 3.2 ± 0.5 mm (0.125 ± 0.020 in.), taking no more than 2 min (Note 13). The amount of hand or finger pressure required will vary greatly, according to the soil. Fragile soils of low plasticity are best rolled under the outer edge of the palm or at the base of the thumb.

NOTE 12—A normal rate of rolling for most soils should be 80 to 90 strokes per minute, counting a stroke as one complete motion of the hand forward and back to the starting position. This rate of rolling may have to be decreased for very fragile soils.

NOTE 13—A 3.2-mm (1/8-in.) diameter rod or tube is useful for frequent comparison with the soil thread to ascertain when the thread has reached the proper diameter, especially for inexperienced operators.

17.1.1 When the diameter of the thread becomes 3.2 mm, break the thread into several pieces. Squeeze the pieces together, knead between the thumb and first finger of each hand, reform into an ellipsoidal mass, and reroll. Continue this alternate rolling to a thread 3.2 mm in diameter, gathering together, kneading and rerolling, until the thread crumbles under the pres-



sure required for rolling and the soil can no longer be rolled into a 3.2-mm diameter thread (See Fig. 7). It has no significance if the thread breaks into threads of shorter length. Roll each of these shorter threads to 3.2 mm in diameter. The only requirement for continuing the test is that they are able to be reformed into an ellipsoidal mass and rolled out again. The operator shall at no time attempt to produce failure at exactly 3.2 mm diameter by allowing the thread to reach 3.2 mm, then reducing the rate of rolling or the hand pressure, or both, while continuing the rolling without further deformation until the thread falls apart. It is permissible, however, to reduce the total amount of deformation for feebly plastic soils by making the initial diameter of the ellipsoidal mass nearer to the required 3.2-mm final diameter. If crumbling occurs when the thread has a diameter greater than 3.2 mm, this shall be considered a satisfactory end point, provided the soil has been previously rolled into a thread 3.2 mm in diameter. Crumbling of the thread will manifest itself differently with the various types of soil. Some soils fall apart in numerous small aggregations of particles, others may form an outside tubular layer that starts splitting at both ends. The splitting progresses toward the middle, and finally, the thread falls apart in many small platy particles. Fat clay soils require much pressure to deform the thread, particularly as they approach the plastic limit. With these soils, the thread breaks into a series of barrel-shaped segments about 3.2 to 9.5 mm ($\frac{1}{4}$ to $\frac{3}{8}$ in.) in length.

17.2 Gather the portions of the crumbled thread together and place in a weighed container. Immediately cover the container.

17.3 Select another 1.5 to 2.0 g portion of soil from the original 20-g specimen and repeat the operations described in 17.1 and 17.2 until the container has at least 6 g of soil.

17.4 Repeat 17.1 through 17.3 to make another container holding at least 6 g of soil. Determine the water content, in percent, of the soil contained in the containers in accordance with Method D 2216. Make all weighings on the same balance.

NOTE 14—The intent of performing two plastic limit trials is to verify the consistency of the test results. It is acceptable practice to perform only one plastic limit trial when the consistency of the test results can be confirmed by other means.

18. Calculations

18.1 Compute the average of the two water contents. If the difference between the two water contents is greater than two percentage points, repeat the test. The plastic limit is the average of the two water contents.

PLASTICITY INDEX

19. Calculations

19.1 Calculate the plasticity index as follows:

$$PI = LL - PL$$

where:

LL = the liquid limit

PL = the plastic limit

Both LL and PL are whole numbers. If either the liquid limit or plastic limit could not be determined, or if the plastic limit is equal to or greater than the liquid limit, report the soil as nonplastic, NP.

20. Report

20.1 Report the following information:

20.1.1 Sample identifying information.

20.1.2 Any special specimen selection process used, such as removal of sand lenses from undisturbed sample.

20.1.3 Report sample as air-dried if the sample was air-dried before or during preparation.

20.1.4 Liquid limit, plastic limit, and plasticity index to the nearest whole number and emitting the percent designation. If the liquid limit or plastic limit tests could not be performed, or if the plastic limit is equal to or greater than the liquid limit, report the soil as nonplastic, NP.

20.1.5 An estimate of the percentage of sample retained on the 425- μ m (No. 40) sieve, and

20.1.6 Procedure by which liquid limit was performed, if it differs from the multipoint method.

21. Precision and Bias

21.1 No interlaboratory testing program has as yet been conducted using this test method to determine multilaboratory precision.

21.2 The within laboratory precision of the results of tests performed by different operators at one laboratory on two soils using Procedure A for the liquid limit is shown in Table 2.

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TABLE 1 Factors for Obtaining Liquid Limit from Water Content and Number of Drops Causing Closure of Groove

A - (Number of Drops)	K - (Factor for Liquid Limit)
20	0.974
21	0.979
22	0.983
23	0.986
24	0.989
25	0.992
26	0.995
27	0.998
28	1.001
29	1.004
30	1.007

TABLE 2 Within Laboratory Precision for Liquid Limit

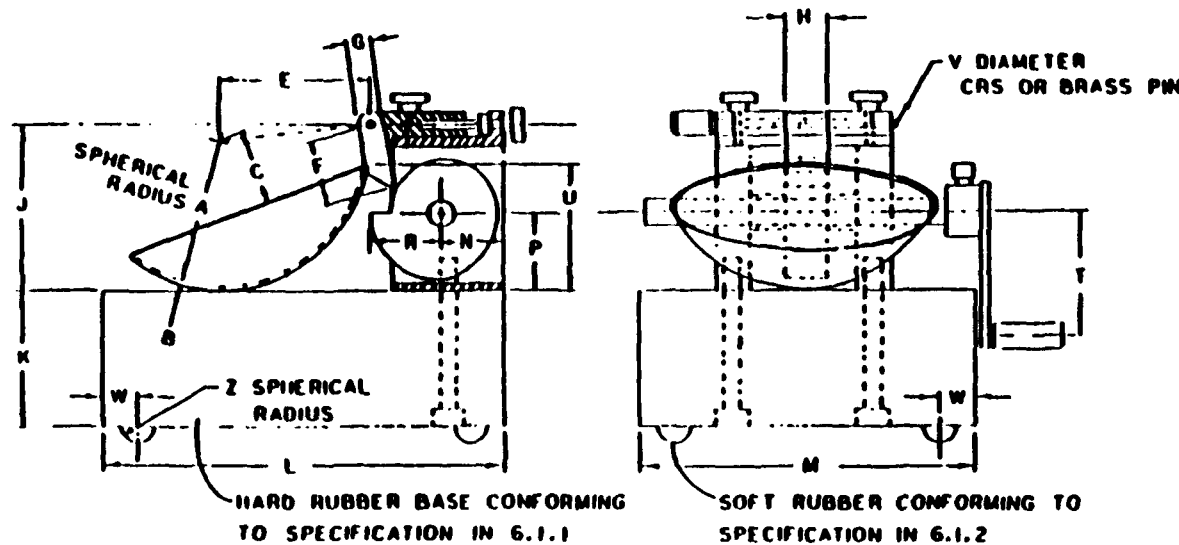
Average Value, %		Standard Deviation, %
Soil A:		
PL	31.9	1.07
LL	37.9	1.07
Soil B:		
PL	30.1	1.21
LL	32.6	0.98

DIMENSIONS

LETTER	A ^a	B ^a	C ^a	E ^a	F	G	H	J ^a	K ^a	L ^a	M ^a
MM	54 ± 0.5	2 ± 0.1	27 ± 0.5	56 ± 2.0	32	10	16	60 ± 1.0	50 ± 2.0	150 ± 2.0	125 ± 2.0
LETTER	N	P	R	T	U ^a	V	W	Z			
MM	24	28	24	45	47 ± 1.0	3.8	13	6.5			

^a ESSENTIAL DIMENSIONS

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CAM ANGLE DEGREES	CAM RADIUS
0	0.742 R
30	0.753 R
60	0.764 R
90	0.773 R
120	0.784 R
150	0.796 R
180	0.818 R
210	0.854 R
240	0.901 R
270	0.945 R
300	0.974 R
330	0.995 R
360	1.000 R

0.418

DIMENSIONS

LETTER	A ^a	B ^a	C ^a	D ^a	E ^a	F ^a
MM	2 ± 0.1	11 ± 0.2	40 ± 0.3	8 ± 0.1	50 ± 0.3	2 ± 0.1
LETTER	G	H	J	K ^a	L ^a	N
MM	10 MINIMUM	13	60	10 ± 0.05	60 DEG ± 1 DEG	20

^a ESSENTIAL DIMENSIONS

^b BACK AT LEAST 15 MM FROM TIP

NOTE: DIMENSION A SHOULD BE 6.9-7.0 AND DIMENSION D SHOULD BE 8.0-8.1 WHEN NEW TO ALLOW FOR ADEQUATE SERVICE LIFE

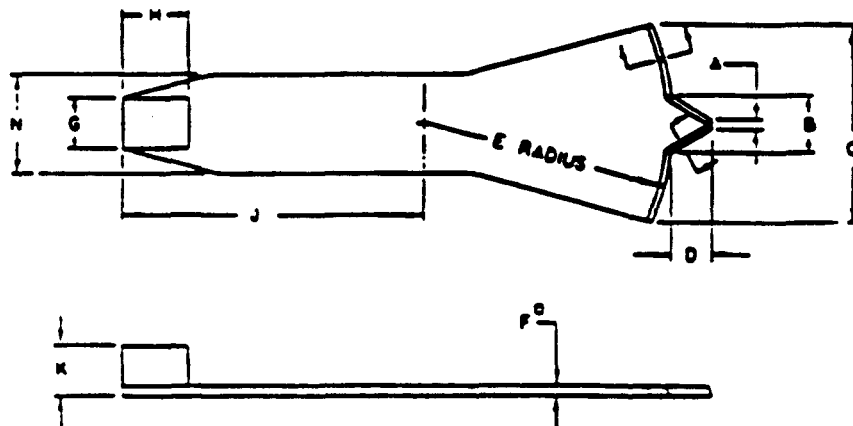
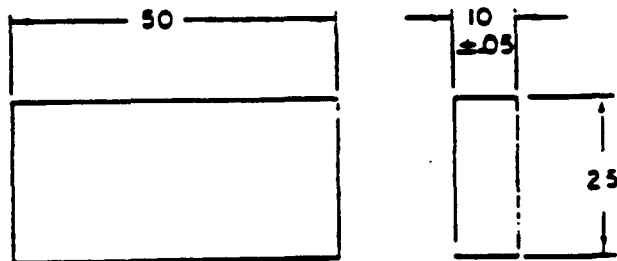


FIG. 1 Growing Tool (Optional Height-of-Drop Cage Attached)



DIMENSIONS IN MILLIMETRES

FIG. 3 Height of Drop Cage

0 4318

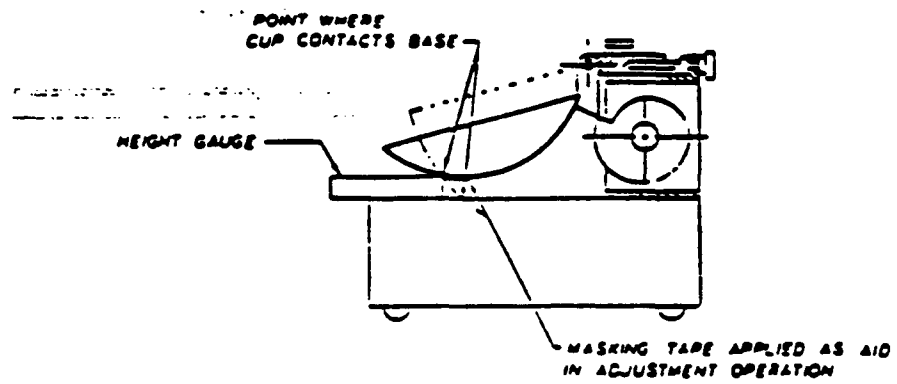


FIG. 4 Calibration for Height of Drop

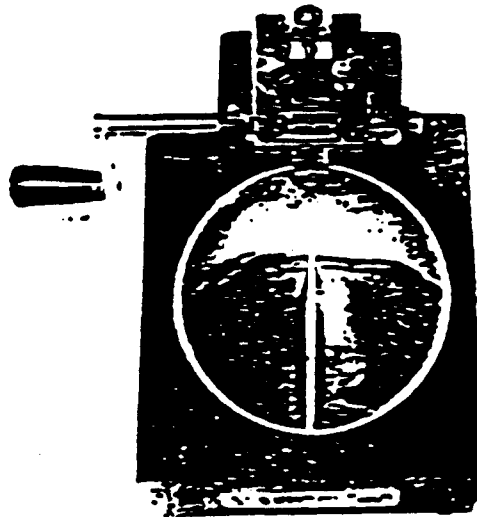


FIG. 5 Grooved Soil Pen in Liquid Lubricant Device

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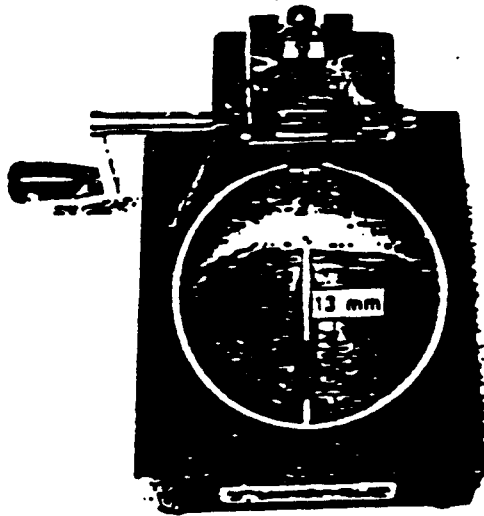


FIG. 6 Soil Pen After Groove Has Closed



FIG. 7 Low Clay Soil at the Plastic Limit

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Standard Test Method for ONE-DIMENSIONAL CONSOLIDATION PROPERTIES OF SOILS¹

This standard is issued under the fixed designation D 2435; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers a procedure for determining the rate and magnitude of consolidation of soil when it is restrained laterally and loaded and drained axially.

NOTE 1—The values stated in SI units are to be regarded as the standard. The values stated in inch-pound units are approximate.

2. Applicable Documents

2.1 ASTM Standards:

D 422 Particle-Size Analysis of Soils²

D 423 Test for Liquid Limit of Soils²

D 424 Test for Plastic Limit and Plasticity Index of Soils²

D 854 Test for Specific Gravity of Soils²

D 1587 Thin-Walled Tube Sampling of Soils²

D 2216 Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures²

D 3550 Ring-Lined Barrel Sampling of Soils²

3. Summary of Method

3.1 The method requires that an element of soil be restrained laterally and axially loaded in increments with a constant stress being applied until all of the excess pore water pressures have dissipated for each increment. During the compression process measurements of decrease in the sample height are made and these data are used to compute the parameters that describe the relationship between the effective stress and void ratio or strain and describe the rate at which compression can occur.

4. Significance and Use

4.1 The compressibility of soils as deter-

mined from this test is one of the most useful properties that can be obtained from laboratory testing. The data from the consolidation test can be used to develop an estimate of both the rate and the amount of both differential and total settlement of a structure or a landfill. Estimates of this type are often of key importance in first selecting a foundation type and secondly in evaluating its adequacy.

5. Apparatus

5.1 *Load Device*—A suitable device for applying vertical loads to the specimen. The device should be capable of maintaining specified loads for long periods of time with an accuracy of $\pm 0.5\%$ of the applied load and should permit application of a given load increment within a period of 2 s. without significant impact.

5.2 *Consolidometer*—A device to hold the specimen in a ring which is either fixed to the base or floating (supported by friction on periphery of specimen) with porous stones on each face of the specimen. The consolidometer shall also provide means for submerging the specimen, for applying a vertical load, and for measuring the change in height of specimen. The consolidometer ring shall conform to the following requirements:

5.2.1 *Minimum Specimen (Ring) Diameter*—The minimum specimen diameter shall be 50 mm (2.00 in.), and shall be at least 5 mm ($\frac{1}{4}$

¹ This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.05 on Structural Properties of Soils.

Current edition approved Oct. 31, 1980. Published December 1980. Originally published as D 2435 - 65 T. Last previous edition D 2435 - 70.

² Annual Book of ASTM Standards, Part 19.

in.) less than the inside diameter of the sample tube if samples are extruded and trimmed.

5.2.2 Minimum Specimen Height—The minimum specimen height shall be 13 mm (0.5 in.) but shall be not less than 10 times the maximum particle diameter.

5.2.3 Minimum Specimen Diameter-to-Height Ratio—The minimum specimen diameter-to-height ratio shall be 2.5.

5.2.4 Rigidity—The rigidity of the ring shall be such that, under hydrostatic stress conditions in the specimen, the change in diameter of the ring will not exceed 0.03 % of the diameter under the greatest load applied.

5.2.5 The ring shall be made of a material that is noncorrosive in relation to the soil tested. The inner surface shall be highly polished or shall be coated with a low-friction material. Silicon grease is recommended or polytetrafluoroethylene for nonsandy soils.

5.3 Porous Stones—The porous stones shall be of silicon carbide, aluminum oxide, or metal that is not attacked by the soil or soil moisture. The grade of the stones shall be fine enough to prevent intrusion of soil into the pores of the stone. If necessary, a filter paper² may be used to prevent intrusion of the soil into the stones. However, the permeability of the stones, and filter paper, if used, must be sufficiently high to prevent retardation of the drainage of the specimen. The stones shall be clean and free of cracks, chips, and nonuniformities.

5.3.1 The diameter of the top stone shall be 0.2 to 0.5 mm (0.01 to 0.02 in.) less than that of the inside diameter of the ring. If a floating ring is used, the bottom stone shall have the same diameter as the top stone. The use of tapered stones is recommended, with the larger diameter in contact with the soil.

5.3.2 The stone thickness shall be sufficient to prevent breaking. The top stone shall be loaded through a corrosion-resistant plate of sufficient rigidity to prevent breakage of the stone.

5.4. Storage—Storage of sealed samples should be such that no moisture is lost during storage, that is, no evidence of partial drying of the ends of the samples or shrinkage. Time of storage should be minimized, particularly when the soil or soil moisture is expected to react with the sample tubes.

5.4.1 Sample Preparation Environment—

Test specimens shall be prepared in an environment where soil moisture change during preparation does not exceed 0.2 %. (A high humidity moisture room is usually used for this purpose.)

5.5 Temperature—Tests should be performed in an environment where temperature fluctuations are less than $\pm 4^{\circ}\text{C}$ (39°F) and there is not any direct contact with sunlight.

5.6 Trimmer, or cylindrical cutter, for trimming the sample down to the inside diameter of the consolidometer ring with a minimum of disturbance. The cutter shall have a highly polished surface and be coated with a low-friction material.

5.7 Balance, sensitive to 0.1 g or to 0.1 % of the total mass of the test specimen.

5.8 Drying Oven, that can be maintained uniformly at $110 \pm 5^{\circ}\text{C}$ ($230 \pm 9^{\circ}\text{F}$). A forced-draft oven is recommended.

5.9 Extensometer, to measure change in height of the specimen with a sensitivity of 0.0025 mm (0.0001 in.).

5.10 Miscellaneous Equipment, including spatulas, knives, wire saws, used in preparing the specimen.

5.11 Moisture Content Containers, shall be in accordance with Method D 2216.

6. Sampling

6.1 Relatively undisturbed samples should be obtained and used for consolidation testing because the meaningfulness of the results diminishes greatly with sample disturbance. Methods D 1587 and D 3550 cover procedures and apparatus that may be used to obtain satisfactory samples for testing.

7. Specimen Preparation (Note 2)

7.1 Prepare the sample in an environment in accordance with 5.4.1. Trim the specimen to the inside diameter of the consolidometer, forcing it directly into the ring during trimming. Trim it flush with the plane surface of the ring. For soft to medium soils, a wire saw should be used for trimming the top and bottom of the specimen to minimize smearing. A straightedge with a sharp cutting edge may be used for the final trim after the excess soil has first been removed with a wire saw. For stiff soils, a sharpened straightedge alone may be used for

² Use a hardened, fast filter paper.

trimming the top and bottom. If desired, the specimen height may be made less than the ring height by partial extrusion and trimming, provided the minimum specimen thickness requirements in 5.2.2 and 5.2.3 are met. A specimen ring with the cutting edge attached provides the most accurate fit in moist soils.

NOTE 2—Precautions should be taken to minimize disturbance of the soil or changes in moisture and density during sample transportation and preparation. Vibration, distortion, and compression must be avoided.

7.2 Fibrous soils, such as peat, and those soils that are easily damaged by trimming, may be transferred directly from the sampling tube to the ring provided that the ring has the same diameter as the sample tube.

7.3 Specimens obtained using a ring-lined sampler may be used without prior trimming, provided they comply with the requirements of Method D 3550 and this method.

7.4 Determine the mass of the specimen in the consolidation ring.

8. Calibration

8.1 Assemble the consolidometer with a copper or hard steel disk of approximately the same height as the test specimen and 1 mm (0.04 in.) smaller diameter than the ring in place of the sample. Moisten the porous stones. If filter papers are to be used (see 5.3), they should be moistened and sufficient time for the moisture to be squeezed from them should be allowed during the calibration process.

8.2 Load and unload the consolidometer as in the test and measure the deformation for each load applied.

8.3 Plot or tabulate the corrections to be applied to the deformation of the test specimen at each load applied. Note that the metal disk will deform also. However, the correction due to this deformation will be negligible for all but extremely stiff soils. If necessary, the compression of the metal disk can be computed and applied to the corrections.

9. Natural Moisture Content, Weight, Volume, Specific Gravity, Liquid and Plastic Limits, and Particle Size Distribution Determinations

9.1 Use the material trimmed adjacent to the specimen (see Section 7) to determine the natural moisture content in accordance with

Method D 2216 and the specific gravity with Method D 854. Determine the initial wet mass of the specimen by subtracting the mass of the specimen ring from the combined mass of the specimen ring and the specimen. Determine the initial volume of the specimen from the diameter and height of the specimen. The value of moisture content determined from the trimmings is approximate but permits determination of the void ratio before the test is complete. The most accurate determination of the specimen dry mass and moisture content is found by drying the entire specimen at the end of the test. This procedure should be used unless a portion of the specimen is needed for index tests (see 10.9). The specific gravity can be estimated where an accurate void ratio is not needed.

9.2 The liquid limit, as determined in accordance with Method D 423, the plastic limit, as determined in accordance with Method D 424, and the particle size distribution, for soils with substantial granular material, as determined in accordance with Method D 422, are useful in identifying the soil and in correlating the results of tests on different soils. It is recommended that liquid and plastic limits be determined on the trimmings or on representative portions of the test specimen if the soil exhibits significant heterogeneity.

10. Procedure

10.1 The objective of the preparation of the stones and other apparatus prior to their contact with the specimen is to prevent change in moisture content of the specimen. Thus, very dry, highly expansive soils should be placed on dry stones but moist partially saturated soils may be placed on stones that have simply been dampened. If the specimen is saturated and is believed not to exhibit a high affinity for water, the porous stones should be boiled after cleaning with a nonabrasive brush and kept saturated until the time of their contact with the specimen. Assemble the ring, specimen and porous stones. With the consolidometer assembled, enclose the specimen, ring, filter paper (when used), and porous stones with a loose-fitting plastic or rubber membrane to prevent change in specimen volume due to evaporation. This step may be omitted if the specimen is to be inundated after the first load increment is applied as provided in 10.8.

10.2 Place the consolidometer in the loading device and apply a seating pressure of 5 kPa (100 lb/ft²). Within 5 min after application of the seating load, adjust the extensometer dial gage for the initial or zero reading. (For very soft soils a seating pressure of 2 or 3 kPa (about 50 lb/ft²) or less is desirable.)

10.3 Place loads on the consolidometer to obtain pressures on the soil of approximately 5, 10, 20, 40, 80, etc., kPa (100, 200, 400, 800, 1600, etc., lb/ft²), with each pressure maintained constant as required in 10.4. (Smaller increments may be desirable on very soft specimens or when it is desirable to determine the preconsolidation pressure with more precision.) Loading of the specimen should continue into the virgin compression region so that the slope and shape of the virgin compression curve may be assessed. Typically, a final pressure that is equal to or greater than four times the preconsolidation pressure of the sample is required for this assessment. In the case of overconsolidated clays in particular, it may be desirable to use an unloading-reloading cycle to better evaluate recompression parameters, but such a procedure is optional.

10.4 For at least two load increments (including at least one load increment after the preconsolidation pressure has been exceeded), record the height or change in height of the specimen before each pressure increment is applied and at time intervals of approximately 0.1, 0.25, 0.5, 1, 2, 4, 8, 15, and 30 min, 1, 2, 4, 8, etc., h, measured from the time of each incremental pressure application. These time-rate readings need be obtained only for saturated specimens. Readings should continue at least until the slope of the characteristic linear secondary compression portion of the thickness versus log of time plot is apparent (see 11.1), unless the method 11.6 is used, in which case the next loading may proceed as soon as 100 % consolidation is completed. (For soils that have slow primary consolidation, pressures should be applied for at least 24 h. In extreme cases, or where secondary compression must be evaluated, they should be applied much longer.) Then apply the next pressure increment. For pressure increments where time versus deformation data are not required, the load should be left on the sample for essentially the same length of time as when time versus de-

mation readings are taken. Sufficient readings should be taken near the end of the pressure-increment period to allow for any needed extrapolation of the time-deformation curve.

10.5 If thickness versus square root of time plots are to be made, the time intervals may be adjusted to those that have easily obtained square roots, for example, 0.09, 0.25, 0.49, 1 min, 4 min, 9 min, etc.

10.6 *Rebound*—Where rebound or unloading characteristics are desired, unload soil by pressure decrements in reverse order. However, if desired, each successive load can be only one fourth as large as the preceding load. Record at time intervals as suggested in 10.4. Note, however, that for most soils the rebound will be complete in less time than would be required for an increment of load during primary consolidation, but sufficient readings should be taken to verify that rebound is essentially complete.

10.7 An alternative loading, unloading, or reloading schedule may be employed that reproduces the construction stress changes, or obtains better definition of some part of the stress-deformation curve, or aids in interpreting the field behavior of the soil. This should be clearly indicated on the test results.

10.8 If the test is performed on an undisturbed sample that was either saturated under field conditions or obtained from below the water table, it should be inundated after the first load increment is applied. As inundation and specimen wetting occur, the load should be increased if required, to prevent the specimen from swelling, unless it tends to swell under the estimated in situ vertical stress. If the specimen compresses after inundation, simply record the amount of compression. Specimens also may be inundated at pressures that simulate future inundation under field conditions. In such cases, the pressure at inundation and any resulting effects, such as expansion or increased compression, should be noted in the test results.

10.9 To minimize swell during disassembly, the specimen should be rebounded back to a very small stress and dismantled quickly after releasing the final load on the specimen. Remove the specimen and the ring from the consolidometer and wipe the free water from the ring and specimen. Remove the specimen from the ring and determine its mass, oven dry it.

and reweigh it to obtain the dry mass of solids and the final water content. If the original soil sample is very heterogeneous, any index tests should be performed on a portion of the test specimen and the remainder can be used for a final water content measurement. Porous stones should be boiled clean after the test to prevent clay from drying on them and reducing their permeability.

11. Calculation

11.1 From those increments of load where time-rate readings are obtained, plot the deformation readings versus the log of time (in minutes) for each increment of load or pressure as the test progresses, and for any increments of rebound where time versus deformation data have been obtained.

11.2 Find the deformation representing 100 % primary consolidation for each load increment. First draw a straight line through the points representing the final readings and that exhibit a straight line trend and a flat slope. Draw a second straight line tangent to the steepest part of the deformation-log time curve. The intersection represents the deformation corresponding to 100 % primary consolidation. Correct the deformation at 100 % consolidation in accordance with the results of the calibration (see Section 8). Compression that occurs subsequent to 100 % primary consolidation is defined as secondary compression.

11.3 Find the deformation representing 0 % primary consolidation by selecting the deformations at any 2 times that have a ratio of 1 to 4. The deformation corresponding to the larger of the two times should be greater than $\frac{1}{4}$ but less than $\frac{1}{2}$ of the total change in deformation for the load increment. The deformation corresponding to 0 % primary consolidation is equal to the deformation corresponding to the smaller time interval less the difference in the deformations for the two selected times.

11.4 The deformation corresponding to 50 % primary consolidation for each load increment is equal to the average of the uncorrected deformations corresponding to the 0 and 100 % deformations. The time required for 50 % consolidation under any load increment may be found graphically from the deformation-log time curve for that load increment by observing the time that corresponds to 50 % of the primary consolidation of the curve.

11.5 For each load increment for which time-rate readings were obtained, compute the coefficient of consolidation, c_v , as follows:

$$c_v = \frac{0.05H^2}{t_{50}}$$

where:

H = sample height in metres (feet) for a doubly drained sample at 50 % consolidation.

t_{50} = time for 50 % consolidation in years, and

c_v = coefficient of consolidation in m^2/year (ft^2/year).

If H is in millimetres and t in seconds or minutes, c_v will be expressed in mm^2/s or mm^2/min , respectively, and conversion to more convenient units is desirable.

11.6 An alternative procedure requiring a plot of deformation versus the square root of time in minutes may be used to determine the points of 0 and 100 % consolidation as well as the coefficient of consolidation for each load increment. The initial part of the curve is approximated by a straight line. The line is extrapolated back to $t = 0$. The corresponding deformation ordinate represents 0 % primary consolidation. A second straight line is drawn through this point so that the abscissa of this line is 1.15 times the abscissa of the straight line approximation of the initial part of the curve. The intersection of the new line with the deformation-square root of time curve corresponds to 90 % primary consolidation. The deformation at 100 % primary consolidation is $\frac{1}{2}$ more than the difference in deformations between 0 and 90 % consolidation. Similarly, the deformation at 50 % consolidation is $\frac{1}{2}$ of the difference between that at 0 and 90 %. The coefficient of consolidation can be found from the time of 90 % consolidation by the formula:

$$c_v = \frac{0.21H^2}{t_{90}}$$

where:

H = sample height for a doubly drained sample at 90 % consolidation, m (or ft).

t_{90} = time for 90 % consolidation, years, and

c_v = coefficient of consolidation, m^2/year (ft^2/year).

If H is in millimetres and t in seconds or minutes, the units of c_v will be mm^2/s or mm^2/min , respectively, and conversion to more convenient units is desirable. The square root

of time method may also be used to obtain a value of t_m if desired.

11.7 Compute initial void ratio, water content, unit weight, and degree of saturation, based on the dry weight of the total specimen. Specimen volume is computed from measurements of the container ring height and diameter. Volume of solids is computed by dividing the dry weight of specimen by the specific gravity of the solids times the unit weight of water. Volume of voids is assumed to be the difference between the specimen volume and the volume of the solids.

11.8 Compute void ratio corresponding to 100 % primary consolidation (using corrected deformation readings) for each load. As an alternative, compute percent compression at 100 % primary consolidation for each load from the initial sample height. As a second alternative, compute void ratios (or values of percent compression) using the deformation values obtained after a selected time interval that shall include some portion of secondary compression, and such time interval shall be the same for each load increment. However, if the "equilibrium" value chosen is other than the 100 % primary consolidation point, a note to this effect should be included with the test results. It should be noted that the second alternative cited above will result in somewhat lower values of the preconsolidation pressure than are obtained when the 100 % primary consolidation points are used.

12. Report

12.1 The report shall include the following information:

12.1.1 Identification and description of the test sample, including whether soil is undisturbed, remolded, compacted, or otherwise prepared.

12.1.2 Initial and final moisture content.

12.1.3 Dry mass and initial and final wet unit weight.

12.1.4 Initial percentage saturation.

12.1.5 Specific gravity of solids or Atterberg Limit gradation data if obtained.

12.1.6 Specimen dimensions.

12.1.7 Condition of test (natural moisture or inundated, pressure at inundation).

12.1.8 Preparation procedure used relative to trimming; state whether specimen was

trimmed, extruded directly into the ring, or tested directly in a ring from a ring-lined sampler.

12.1.9 Plot of log of time or square root of time versus deformation, for those load increments where time-rate readings were taken. For organic or highly micaceous soils, or other soils with appreciable secondary compression, it is highly desirable that log of time plots extending into the secondary compression region be included.

12.1.10 Plot of void ratio versus log of pressure curve or percent compression versus log of pressure curve.

12.1.11 In cases where time rate of consolidation readings have been taken for several load increments, a plot of coefficient of consolidation versus log of average pressure. As an alternative, a plot of log of coefficient of consolidation versus log of average pressure curve may be used. The method used for computing c_v should be noted. If time-rate readings were obtained for only two load increments, simply tabulate the values of c_v versus the average pressure for the increment.

12.1.12 All departures from the procedure outlined, including special loading sequences. For example, it may be desirable to inundate and load the specimen precisely in accordance with the wetting or loading pattern expected in the field in order to best simulate the response. Smaller than standard load increment ratios may be desirable for soils that are highly sensitive or whose response is highly dependent on strain rate.

13. Precision and Accuracy

13.1 Section 5 of this method specifies the sensitivity of the load and deformation measurements. The corresponding accuracy of the applied stress and resultant strain in the specimen can be computed from the specimen dimensions. The accuracy with which the test results can be applied to the field varies from case to case and depends on:

13.1.1 The quality of samples used.

13.1.2 The number of samples tested.

13.1.3 The vertical and horizontal distribution of the samples tested, and

13.1.4 The heterogeneity of the soil profile in the field.

13.2 A statistical analysis that includes both

the sampling program and the laboratory test results is required to assess, in any meaningful way, the accuracy with which the test results apply to the field. In the absence of such an analysis, most engineers assess the applicability of the test results to the field qualitatively by

looking at the variation in the test results and the variations in the boring logs. In general, it can be noted that the accuracy of settlement predictions based on consolidation tests increase (on a percentage basis) with the compressibility of the soil.

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Standard Test Method for UNCONSOLIDATED, UNDRAINED COMPRESSIVE STRENGTH OF COHESIVE SOILS IN TRIAXIAL COMPRESSION¹

This standard is issued under the fixed designation D 2850; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the unconsolidated, undrained compressive strength (or maximum principal stress difference) of cylindrical specimens of cohesive soils in undisturbed, remolded, or compacted conditions using constant rate of deformation (strain-controlled) application of the axial compression test load and where the specimen is subjected to a confining fluid pressure in a triaxial chamber. No drainage of the specimen is permitted during the test. The test method provides for the measurement of the total stresses applied to the specimen, that is, the stresses are not corrected for pore-water pressure. The total stress is the sum of the effective stress and the pore pressure.

1.2 This test method provides data for determining undrained strength properties and stress-strain relations for soils.

NOTE 1—The determination of the unconsolidated, undrained strength of cohesive soils without lateral confinement is covered by Test Methods D 2166.

NOTE 2—This test method does not provide a procedure for back pressure saturation of the test specimens. If back pressure saturation of the specimens is required, the test must be performed utilizing procedures and apparatus similar to those required for a consolidated undrained triaxial test. However, due to consolidation, which could occur during the saturation phase, this modified procedure is not truly unconsolidated. A test method for the consolidated undrained triaxial test is currently under development in Subcommittee D18.05.

NOTE 3—This test method does not include a procedure for obtaining pore pressure measurements. Furthermore, at the rapid strain rates used in this test method such measurements could be inaccurate. If pore pressure measurements are desired, alternative procedures such as the U.S. Bureau of Reclamation Method E-17 can be used.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 422 Method for Particle-Size Analysis of Soils²
- D 653 Terms and Symbols Relating to Soil and Rock²
- D 854 Test Method for Specific Gravity of Soils²
- D 1587 Practice for Thin-Walled Tube Sampling of Soils²
- D 2166 Test Methods for Unconfined Compressive Strength of Cohesive Soil²
- D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures²
- D 2487 Test Method for Classification of Soils for Engineering Purposes²
- D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)²

¹ This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.05 on Structural Properties of Soils.

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² Annual Book of ASTM Standards, Vol 04.08.

D4220 Practices for Preserving and Transporting Soil Samples²

D4318 Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils²

3. Terminology

3.1 Definitions:

3.1.1 *triaxial compression test*—a test in which a cylindrical specimen of soil encased in an impervious membrane is subjected to a confining pressure and then loaded axially to failure in compression (as defined in 3.2.1).

3.1.2 *principal stress difference or deviator stress*—the difference between the major and minor principal stresses in a triaxial test.

3.1.2.1 *Discussion*—The principal stress difference or deviator stress is equal to the axial load applied to the specimen divided by the corrected cross-sectional area of the specimen, as prescribed in Section 8. The major principal stress in the specimen is equal to the deviator stress plus the chamber pressure, and the minor principal stress in the specimen is equal to the chamber pressure.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *failure*—the failure stresses are taken as the stresses in the specimen corresponding to the maximum principal stress difference (deviator stress) attained or the principal stress difference (deviator stress) at 15% axial strain, whichever is obtained first during the performance of a test.

3.2.2 *unconsolidated-undrained compressive strength*—the value of the maximum principal stress difference or deviator stress obtained during the test.

4. Significance and Use

4.1 In this test method, the compressive strength of a soil is determined in terms of the total stress, therefore, the resulting strength depends on the pressure developed in the pore fluid during loading. In this test method, fluid flow is not permitted from or into the soil specimen as the load is applied, therefore the resulting pore pressure, and hence strength, differs from that developed in the case where drainage can occur.

4.2 If the test specimens are 100% saturated, consolidation cannot occur when the confining pressure is applied nor during the shear portion of the test since drainage is not permitted. Therefore, if several specimens of the same material

are tested, and if they are all at approximately the same water content and void ratio when they are tested, they will have approximately the same undrained shear strength. The Mohr failure envelope will usually be a horizontal straight line over the entire range of confining stresses applied to the specimens if the specimens are fully saturated.

4.3 If the test specimens are partially saturated or compacted specimens, where the degree of saturation is less than 100%, consolidation may occur when the confining pressure is applied and during shear, even though drainage is not permitted. Therefore, if several partially saturated specimens of the same material are tested at different confining stresses, they will not have the same undrained shear strength. Thus, the Mohr failure envelope for unconsolidated undrained triaxial tests on partially saturated soils is usually curved.

4.4 The unconsolidated undrained triaxial strength is applicable to certain design situations in geotechnical engineering practice where the loads are assumed to take place so rapidly that there is insufficient time for the induced pore-water pressure to dissipate and for consolidation to occur during the loading period (that is, drainage does not occur). The unconsolidated undrained triaxial strength is used to determine strengths at the end of construction.

4.5 Compressive strengths determined using this procedure may not apply in cases where the loading conditions in the field differ significantly from those used in this test method.

5. Apparatus

5.1 *Axial Loading Device*—The axial compression device may be screw jack driven by an electric motor through a geared transmission, a hydraulic or pneumatic loading device, or any other compression device with sufficient capacity and control to provide the rate of loading prescribed in 7.5. When the loading device is set to advance at a certain rate of strain, the actual rate of strain shall not deviate by more than $\pm 10\%$. Vibrations due to the operation of the loading device shall be kept at a minimum.

NOTE 4—A loading device may be said to provide sufficiently small vibrations if there are no visible ripples in a glass of water placed on the loading platen when the device is operating at the speed at which the test is performed.

5.2 Axial Load-Measuring Device—The axial load-measuring device shall be a load ring, electronic load cell, hydraulic load cell, or any other load-measuring device capable of the accuracy prescribed in this section and may be a part of the axial loading device. The axial load-measuring device shall be capable of measuring the axial load to an accuracy of 1 % of the estimated axial load at failure.

5.3 Chamber Pressure-Maintaining and Measurement Device—The chamber pressure-maintaining and measurement device shall be capable of applying and controlling the chamber pressure to within ± 1 % of the applied chamber pressure. This device may consist of a reservoir connected to the triaxial chamber and partially filled with the chamber fluid (usually water), with the upper part of the reservoir connected to a compressed gas supply; the gas pressure being controlled by a pressure regulator and measured by a pressure gage, electronic pressure transducer, or any other device capable of measuring to the prescribed tolerance. However, a hydraulic system pressurized by deadweight acting on a piston or any other pressure-maintaining and measurement device capable of applying and controlling the chamber pressure to the tolerance prescribed in this section may be used.

5.4 Triaxial Compression Chamber—An apparatus shall be provided in which the cylindrical specimen, enclosed by a membrane sealed to the specimen cap and base, may be placed and subjected to a constant hydrostatic fluid pressure. The apparatus shall include a bushing and piston, aligned with the axis of the specimen, through which the load from the axial loading device is transmitted to the specimen axially between the specimen cap and base. The bushing and piston shall be designed to minimize friction and lateral thrust to the specimen cap.

5.5 Specimen Cap and Base—An impermeable rigid cap and base shall be used to prevent drainage of the specimen. The specimen cap and base shall be constructed of a noncorrosive impermeable material, and each shall have a circular plane surface of contact with the specimen and a circular cross section. The weight of the specimen cap shall produce an axial stress on the specimen of less than 1 kN/m^2 . The diameter of the cap and base shall be equal to the initial diameter of the specimen. The specimen base shall be coupled to the triaxial compression

chamber so as to prevent lateral motion or tilting and the specimen cap shall be designed to receive the piston such that the piston-to-cap contact area is concentric with the cap. The specimen cap during shear shall not tilt more than 5° . The cylindrical surface of the specimen base and cap that contacts the membrane to form a seal shall be smooth and free of scratches.

NOTE 5—The stress produced by the specimen cap can exceed 1 kN/m^2 provided the test data is corrected for the effects of that stress.

5.6 Deformation Indicator—The deformation indicator shall be a dial indicator capable of measuring axial deformation to within 0.03 % of the specimen height and having a travel range of at least 20 % of the initial height of the test specimen, or any other measuring device, such as electronic deformation measuring devices, meeting these requirements of readability and range.

5.7 Rubber Membranes—The rubber membrane used to encase the specimen shall provide reliable protection against leakage. Membranes shall be carefully inspected prior to use, and if any flaws or pinholes are evident, the membrane shall be discarded. In order to offer minimum restraint to the specimen, the unstretched membrane diameter shall be between 90 and 95 % of that of the specimen. The membrane thickness shall not exceed $1/7$ of the diameter of the specimen. The membrane shall be sealed to the specimen base and cap by any method that will produce a positive seal. An equation for correcting the principal stress difference (deviator stress) for the effect of the stiffness of the membrane is given in 8.5.

NOTE 6—The membrane is typically sealed using O-rings with silicon grease between the cap and base and the membrane.

5.8 Sample Extruder—The sample extruder shall be capable of extruding the soil core from the sampling tube in the same direction of travel in which the sample entered the tube and with minimum disturbance of the sample. If the soil core is not extruded vertically, care should be taken to avoid bending stresses on the core due to gravity. Conditions at the time of sample removal may dictate the direction of removal, but the principal concern is to keep the degree of disturbance minimal.

5.9 Specimen Size Measurement Devices—Devices used to measure the height and diameter

of the specimen shall be capable of measuring the desired dimension to within 0.1 % of its actual length and shall be constructed such that their use will not disturb the specimen.

5.10 Timer—A timing device indicating the elapsed testing time to the nearest 1 s shall be used for establishing the rate of strain application prescribed in 7.5.

5.11 Balances—The balance used to weigh specimens shall determine the mass of the specimens to within 0.1 % of the total mass.

5.12 Apparatus for Water Content, as specified in Method D 2216.

5.13 Miscellaneous Apparatus—Specimen trimming and carving tools, membrane and O-ring expanders, compaction apparatus, and data sheets as required.

6. Test Specimens

6.1 Specimen Size—Specimens shall have a minimum diameter of 30 mm and the largest particle contained within the test specimen shall be smaller than $\frac{1}{4}$ of the specimen diameter. If, after completion of a test, it is found that oversize particles are present, indicate this information in the report of test data under remarks. Determine the average height and diameter of the test specimen using the apparatus specified in 5.9. Take a minimum of three height measurements (120° apart) and at least three diameter measurements at each of the quarter points of the height. The height-to-diameter ratio of the specimen shall be between 2 and 2.5.

NOTE 7—If large soil particles are found in the specimen after testing, a particle-size analysis in accordance with Method D 422 may be performed to confirm the visual observation and the results provided with the test report.

6.2 Undisturbed Specimens—Prepare undisturbed specimens from large undisturbed samples or from samples secured in accordance with Practice D 1587 or other acceptable undisturbed tube sampling procedures. Undisturbed samples shall be preserved and transported as outlined for Groups C or D samples in Practices D 4220. Specimens obtained by tube sampling may be tested without trimming, except for the squaring of ends, provided soil characteristics are such that no significant disturbance results from sampling and the specimen is uniformly circular. Handle specimens carefully to minimize disturbance, changes in cross section, or loss of water content.

If compression or any type of noticeable disturbance would be caused by the extrusion device, split the sample tube lengthwise or cut it off in small sections to facilitate removal of the specimen with minimum disturbance. Prepare trimmed specimens in an environment where the change in the water content of the soil is minimized (Note 8). Specimens shall be of uniform, circular cross section perpendicular to the axis of the specimen. Where pebbles or crumbling result in excessive irregularity along the outside edges of the specimen or at the ends, pack soil from the trimmings in the irregularities to produce the desired surface. As an alternative, the ends of the specimen may be capped with a minimal thickness of plaster of paris, hydrostone, or similar material. Where soil conditions permit, a vertical lathe accommodating the total sample may be used as an aid in trimming the specimen to the required diameter. Determine the mass and dimensions of the test specimen in accordance with 5.9 and 5.11. If the specimen is to be capped, determine its mass and dimensions before capping. Enclose the specimen in the rubber membrane and seal the membrane to the specimen base and cap immediately after preparation.

NOTE 8—A controlled high-humidity room is usually used for this purpose.

6.3 Remolded Specimens—Prepare the specimen by first thoroughly working the undisturbed specimen, which has been tested and is still enclosed in the rubber membrane, with the fingers. Then reform the specimen by forming within a mold having dimensions such that the remolded specimen dimensions will be equal to those of the undisturbed specimen. Exercise care to avoid entrapping air in the specimen. This will aid in obtaining a uniform unit weight, in remolding to the same void ratio as the undisturbed specimen, and in preserving the natural water content of the soil.

6.4 Compacted Specimens—Prepare specimens using the compaction method, predetermined water content, and unit weight prescribed by the individual assigning the test. Compacted specimens may be prepared by compacting material in at least six layers, using a pressing or kneading action, into a split mold of circular cross section having dimensions meeting the requirements of 6.1. Material required for the specimen shall be batched by thoroughly mixing soil with sufficient water to produce the desired water

content. After batching, store the material in a covered container for at least 16 h prior to compaction. Specimens may be molded to the desired density by either: (1) kneading or tamping each layer until the accumulative weight of the soil placed in the mold is compacted to a known volume or (2) by adjusting the number of layers, the number of tamps per layer, and the force per tamp. Scarify the top of each layer prior to the addition of material for the next layer. The tamper used to compact the material shall have an area in contact with the soil equal to or less than $\frac{1}{2}$ the area of the mold. After a specimen is formed, with the ends perpendicular to the longitudinal axis, remove the mold and determine the mass and dimensions of the specimen using the devices described in 5.9 and 5.11. Perform one or more water content determinations on excess material used to prepare the specimen in accordance with Method D 2216.

NOTE 9—It is common for the unit weight of the specimen after removal from the mold to be less than the value based on the volume of the mold. This occurs as a result of the specimen swelling after removal of the lateral confinement due to the mold.

NOTE 10—Experience indicates that it is difficult to compact, handle, and obtain valid results with specimens that have a degree of saturation that is greater than about 90 %.

7. Procedure

7.1 Place the membrane on the membrane expander or, if it is rolled onto the specimen, roll the membrane onto the cap or base. Place the specimen on the base. Place the rubber membrane around the specimen and seal it at the cap and base with O-rings or other positive seals at each end. A thin coating of silicon grease on the vertical surfaces of the cap or base will aid in sealing the membrane.

7.2 With the specimen encased in the rubber membrane, which is sealed to the specimen cap and base and positioned in the chamber, assemble the triaxial chamber. Bring the axial load piston into contact with the specimen cap several times to permit proper seating and alignment of the piston with the cap. When the piston is brought into contact the final time, record the reading on the deformation indicator. During this procedure, take care not to apply an axial stress to the specimen exceeding approximately 0.5 % of the estimated compressive strength. If the weight of the piston is sufficient to apply an

axial stress exceeding approximately 0.5 % of the estimated compressive strength, lock the piston in place above the specimen cap after checking the seating and alignment and keep locked until application of the chamber pressure.

7.3 Place the chamber in position in the axial loading device. Be careful to align the axial loading device, the axial load-measuring device, and the triaxial chamber to prevent the application of a lateral force to the piston during testing. Attach the pressure-maintaining and measurement device and fill the chamber with the confining liquid. Adjust the pressure-maintaining and measurement device to the desired chamber pressure and apply the pressure to the chamber fluid. Wait approximately 10 min after the application of chamber pressure before continuing the test.

NOTE 11—In some cases the chamber will be filled and the chamber pressure applied before placement in the axial loading device.

NOTE 12—Make sure the piston is locked or held in place by the axial loading device before applying the chamber pressure.

NOTE 13—The purpose of the waiting period is to allow the specimen to stabilize under the chamber pressure prior to application of the axial load.

7.4 If the axial load-measuring device is located outside of the triaxial chamber, the chamber pressure will produce an upward force on the piston that will react against the axial loading device. In this case, start the test with the piston slightly above the specimen cap, and before the piston comes in contact with the specimen cap, either: (1) measure and record the initial piston friction and upward thrust of the piston produced by the chamber pressure and later correct the measured axial load, or (2) adjust the axial load-measuring device to compensate for the friction and thrust. If the axial load-measuring device is located inside the chamber, it will not be necessary to correct or compensate for the uplift force acting on the axial loading device or for piston friction. In both cases record the initial reading on the deformation indicator when the piston contacts the specimen cap.

7.5 Apply the axial load to produce axial strain at a rate of approximately 1 %/min for plastic materials and 0.3 %/min for brittle materials that achieve maximum deviator stress at approximately 3 to 6 % strain. At these rates, the elapsed time to reach maximum deviator stress will be approximately 15 to 20 min. Continue

the loading to 15 % axial strain, except loading may be stopped when the deviator stress has peaked then dropped 20 % or the axial strain has reached 5 % beyond the strain at which the peak in deviator stress occurred.

7.6 Record load and deformation values at about 0.1, 0.2, 0.3, 0.4, and 0.5 % strain; then at increments of about 0.5 % strain to 3 %; and, thereafter at every 1 %. Take sufficient readings to define the stress-strain curve; hence, more frequent readings may be required in the early stages of the test and as failure is approached.

NOTE 14—Alternate intervals for the readings may be used provided sufficient points are obtained to define the stress-strain curve.

7.7 After completion of the tests, remove the test specimen from the chamber. Determine the water content of the test specimen in accordance with Method D 2216 using the entire specimen, unless representative cuttings are obtained for this purpose, as in the case of undisturbed specimens. Indicate on the test report whether the water content sample was obtained before or after the shear test, as required in 9.1.2.

7.8 Make a sketch, or take a photo, of the test specimen at failure and show the slope angle of the failure surface if the angle is visible and measurable.

8. Calculations

8.1 Calculate the axial strain, ϵ (expressed as a decimal), for a given applied axial load, as follows:

$$\epsilon = \Delta L / L_0$$

where:

ΔL = change in length of specimen as read from deformation indicator, and

L_0 = initial length of test specimen minus any change in length prior to loading.

8.2 Calculate the average cross-sectional area,

1. for a given applied axial load as follows:

$$A = A_0 / (1 - \epsilon)$$

where:

A_0 = initial average cross-sectional area of the specimen, and

ϵ = axial strain for the given axial load (expressed as a decimal).

NOTE 15—In the event that the application of the chamber pressure results in a change in the specimen length, A_0 should be corrected to reflect this change in volume. Frequently, this is done by assuming that

lateral strains are equal to vertical strains. The diameter after volume change would be given by $D = D_0(1 - \Delta L / L_0)$.

8.3 Calculate the principal stress difference (deviator stress), $\sigma_1 - \sigma_3$, for a given applied axial load as follows:

$$\sigma_1 - \sigma_3 = P / A$$

where:

P = measured applied axial load (corrected for uplift and piston friction, if required see 7.4), and

A = corresponding average cross-sectional area.

8.4 *Stress-Strain Curve*—Prepare a graph showing the relationship between principal stress difference (deviator stress) and axial strain, plotting deviator stress as ordinate and axial strain (in percent) as abscissa. Select the compressive strength and axial strain at failure in accordance with the definitions in 3.2.1 and 3.2.2.

8.5 *Correction of Strength Due to Stiffness of Rubber Membrane*—Assuming units are consistent, the following equation, or other acceptable equations, shall be used to correct the principal stress difference or deviator stress for the effect of the rubber membrane if the error in principal stress difference due to the stiffness of the membrane exceeds 5 %:

$$\Delta(\sigma_1 - \sigma_3) = \frac{4t_m\epsilon_1}{D}$$

where:

$\Delta(\sigma_1 - \sigma_3)$ = correction to be subtracted from the measured principal stress difference.

$D = \sqrt{\frac{4A}{\pi}}$ = diameter of specimen.

E_m = Young's modulus for the membrane material.

t = thickness of the membrane, and

ϵ_1 = axial strain.

8.5.1 The Young's modulus of the membrane material may be determined by hanging a 10.0-mm wide strip of membrane over a thin rod, placing another rod along the bottom of the hanging membrane, and measuring the force per unit strain obtained by stretching the membrane. The modulus value may be computed using the following equation assuming units are consistent:

$$E_m = \frac{FL}{A_m \Delta L}$$

where:

- E_m = Young's modulus of the membrane material,
 F = force applied to stretch the membrane,
 A_m = twice the initial thickness of the membrane multiplied by the width of the membrane strip,
 L = unstretched length of the membrane, and
 ΔL = change in length of the membrane due to application of F .

A typical value of E_m for latex membrane is 1400 kN/m².

NOTE 16—The effect of the stiffness of the membrane on the lateral stress is usually assumed to be negligible.

NOTE 17—The correction for rubber membranes is based on simplified assumptions concerning their behavior during shear. Their actual behavior is complex and there is not a consensus on more exact corrections.

8.6 Calculate the major and minor principal total stresses at failure as follows:

- σ_3 = minor principal total stress = chamber pressure, and
 σ_1 = major principal total stress = deviator stress at failure plus chamber pressure.

8.7 Calculate the initial degree of saturation of the test specimen using the initial mass and dimensions.

NOTE 18—The specific gravity determined in accordance with Test Method D 854 is required for calculation of the degree of saturation, or an assumed value may be used provided it is noted in the test report that an assumed value was used.

9. Report

9.1 The report shall include the following:

9.1.1 Identification and visual description of specimen, including soil group name, symbol, whether specimen is undisturbed, remolded, or compacted, and the like. Also include specimen identifying information, such as project, location, boring number, sample number, depth, and the like. Visual descriptions shall be made in accordance with Practice D 2488.

9.1.2 Initial dry unit weight and water content (specify if the water content specimen was ob-

tained before or after the shear and from cuttings or the entire specimen).

9.1.3 Degree of saturation.

9.1.4 Height and diameter of the specimen.

9.1.5 Height to diameter ratio.

9.1.6 The value of the compressive strength and the values of the minor and major principal stresses at failure.

9.1.7 Stress-strain curve as described in 8.4.

9.1.8 Axial strain at failure, in percent.

9.1.9 Average rate of axial strain to failure, percent per minute.

9.1.10 Liquid and plastic limits, if determined, in accordance with Test Method D 4318.

9.1.11 Sketch or photo showing type of failure, that is, bulge, diagonal shear, and the like.

9.1.12 Particle-size analysis, if determined, in accordance with Method D 422.

9.1.13 If a membrane correction was used, the report shall state that a membrane correction was used to adjust the compressive strength and must indicate the membrane correction equation that was used.

9.1.14 In a remarks section note any unusual conditions or other data that would be considered necessary to properly interpret the results obtained, for example, slickensides, stratification, shells, pebbles, roots, or brittleness.

10. Precision and Bias

10.1 No method presently exists to evaluate the precision of a group of triaxial compression tests on undisturbed specimens, due to specimen variability. Undisturbed soil specimens from apparently homogeneous soil deposits at the same location often exhibit significantly different strength and stress-strain properties.

10.2 A suitable test material and method of specimen preparation have not been developed for the determination of laboratory variances of compacted specimens due to the difficulty in producing identical cohesive soil specimens. No estimates of precision for this test method are available.

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Designation: D 2434 - 68 (Reapproved 1974)¹

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Standard Test Method for PERMEABILITY OF GRANULAR SOILS (CONSTANT HEAD)¹

This standard is issued under the fixed designation D 2434; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Section 2 was added editorially and subsequent sections renumbered in July 1964.

1. Scope

1.1 This test method covers the determination of the coefficient of permeability by a constant-head method for the laminar flow of water through granular soils. The procedure is to establish representative values of the coefficient of permeability of granular soils that may occur in natural deposits as placed in embankments, or when used as base courses under pavements. In order to limit consolidation influences during testing, this procedure is limited to disturbed granular soils containing not more than 10 % soil passing the 75- μ m (No. 200) sieve.

2. Applicable Documents

2.1 ASTM Standards:

D422 Method for Particle-Size Analysis of Soils²

D2049 Test Method for Relative Density of Cohesionless Soils³

3. Fundamental Test Conditions

3.1 The following ideal test conditions are prerequisites for the laminar flow of water through granular soils under constant-head conditions:

3.1.1 Continuity of flow with no soil volume change during a test,

3.1.2 Flow with the soil voids saturated with water and no air bubbles in the soil voids,

3.1.3 Flow in the steady state with no changes in hydraulic gradient, and

3.1.4 Direct proportionality of velocity of flow with hydraulic gradients below certain values, at which turbulent flow starts.

3.2 All other types of flow involving partial saturation of soil voids, turbulent flow, and un-

steady state of flow are transient in character and yield variable and time-dependent coefficients of permeability; therefore, they require special test conditions and procedures.

4. Apparatus

4.1 *Permeameters*, as shown in Fig. 1, shall have specimen cylinders with minimum diameters approximately 8 or 12 times the maximum particle size in accordance with Table 1. The permeameter should be fitted with: (1) a porous disk or suitable reinforced screen at the bottom with a permeability greater than that of the soil specimen, but with openings sufficiently small (not larger than 10 % finer size) to prevent movement of particles; (2) manometer outlets for measuring the loss of head, h , over a length, l , equivalent to at least the diameter of the cylinder; (3) a porous disk or suitable reinforced screen with a spring attached to the top, or any other device, for applying a light spring pressure of 22 to 45-N (5 to 10-lbf) total load, when the top plate is attached in place. This will hold the placement density and volume of soil without significant change during the saturation of the specimen and the permeability testing to satisfy the requirement prescribed in 3.1.1.

4.2 *Constant-Head Filter Tank*, as shown in Fig. 1, to supply water and to remove most of

¹ This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.04 on Hydrologic Properties of Soil and Rock.

Current edition approved Sept. 13, 1968. Originally issued 1945. Replaces D 2434 - 63 T.

² Annual Book of ASTM Standards, Vol 04.02.

³ Discontinued—See 1982 Annual Book of ASTM Standards, Part 19.

the air from tap water, fitted with suitable control valves to maintain conditions described in 3.1.2.

NOTE 1—De-aired water may be used if preferred.

4.3 *Large Funnel*, fitted with special cylindrical spouts 25 mm (1 in.) in diameter for 9.5-mm ($\frac{3}{8}$ -in.) maximum size particles and 13 mm ($\frac{1}{2}$ in.) in diameter for 2.00-mm (No. 10) maximum size particles. The length of the spout should be greater than the full length of the permeability chamber—at least 150 mm (6 in.).

4.4 *Specimen Compaction Equipment*²—Compaction equipment as deemed desirable may be used. The following are suggested: a vibrating tamper fitted with a tamping foot 51 mm (2 in.) in diameter; a sliding tamper with a tamping foot 51 mm (2 in.) in diameter; and a rod for sliding weights of 100 g (0.25 lb) (for sands) to 1 kg (2.25 lb) (for soils with a large gravel content), having an adjustable height of drop to 102 mm (4 in.) for sands and 203 mm (8 in.) for soils with large gravel contents.

4.5 *Vacuum Pump or Water-Faucet Aspirator*, for evacuating and for saturating soil specimens under full vacuum (see Fig. 2).

4.6 *Manometer Tubes*, with metric scales for measuring head of water.

4.7 *Balance*, of 2-kg (4.4-lb) capacity, sensitive to 1 g (0.002 lb).

4.8 *Scoop*, with a capacity of about 100 g (0.25 lb) of soil.

4.9 *Miscellaneous Apparatus*—Thermometers, clock with sweep second hand, 250-mL graduate, quart jar, mixing pan, etc.

5. Sample

5.1 A representative sample of air-dried granular soil, containing less than 10 % of the material passing the 75- μ m (No. 200) sieve and equal to an amount sufficient to satisfy the requirements prescribed in 5.2 and 5.3; shall be selected by the method of quartering.

5.2 A sieve analysis (See Method D 422) shall be made on a representative sample of the complete soil prior to the permeability test. Any particles larger than 19 mm ($\frac{3}{4}$ in.) shall be separated out by sieving (Method D 422). This oversize material shall not be used for the permeability test, but the percentage of the oversize material shall be recorded.

NOTE 2—In order to establish representative values of coefficients of permeabilities for the range that may

exist in the situation being investigated, samples of the finer, average, and coarser soils should be obtained for testing.

5.3 From the material from which the oversize has been removed (see 5.2), select by the method of quartering, a sample for testing equal to an amount approximately twice that required for filling the permeameter chamber.

6. Preparation of Specimens

6.1 The size of permeameter to be used shall be as prescribed in Table 1.

6.2 Make the following initial measurements in centimetres or square centimetres and record on the data sheet (Fig. 3); the inside diameter, D , of the permeameter; the length, L , between manometer outlets; the depth, H_1 , measured at four symmetrically spaced points from the upper surface of the top plate of the permeability cylinder to the top of the upper porous stone or screen temporarily placed on the lower porous plate or screen. This automatically deducts the thickness of the upper porous plate or screen from the height measurements used to determine the volume of soil placed in the permeability cylinder. Use a duplicate top plate containing four large symmetrically spaced openings through which the necessary measurements can be made to determine the average value for H_1 . Calculate the cross-sectional area, A , of the specimen.

6.3 Take a small portion of the sample selected as prescribed in 5.3 for water content determinations. Record the weight of the remaining air-dried sample (see 5.3), W_1 , for unit weight determinations.

6.4 Place the prepared soil by one of the following procedures in uniform thin layers approximately equal in thickness after compaction to the maximum size of particle, but not less than approximately 15 mm (0.60 in.).

6.4.1 For soils having a maximum size of 9.5 mm ($\frac{3}{8}$ in.) or less, place the appropriate size of funnel, as prescribed in 4.3, in the permeability device with the spout in contact with the lower porous plate or screen, or previously formed layer, and fill the funnel with sufficient soil to form a layer, taking soil from different areas of the sample in the pan. Lift the funnel by 15 mm (0.60 in.), or approximately the unconsolidated layer thickness to be formed, and spread the soil with a slow spiral motion, working from the perimeter of the device toward the center, so that

a uniform layer is formed. Remix the soil in the pan for each successive layer to reduce segregation caused by taking soil from the pan.

6.4.2 For soils with a maximum size greater than 9.5 mm ($\frac{3}{8}$ in.), spread the soil from a scoop. Uniform spreading can be obtained by sliding a scoopful of soil in a nearly horizontal position down along the inside surface of the device to the bottom or to the formed layer, then tilting the scoop and drawing it toward the center with a single slow motion; this allows the soil to run smoothly from the scoop in a windrow without segregation. Turn the permeability cylinder sufficiently for the next scoopful, thus progressing around the inside perimeter to form a uniform compacted layer of a thickness equal to the maximum particle size.

6.5 Compact successive layers of soil to the desired relative density by appropriate procedures, as follows, to a height of about 2 cm (0.8 in.) above the upper manometer outlet.

6.5.1 *Minimum Density (0 % Relative Density)*—Continue placing layers of soil in succession by one of the procedures described in 6.4.1 or 6.4.2 until the device is filled to the proper level.

6.5.2 *Maximum Density (100 % Relative Density)*:

6.5.2.1 *Compaction by Vibrating Tamper*—Compact each layer of soil thoroughly with the vibrating tamper, distributing the light tamping action uniformly over the surface of the layer in a regular pattern. The pressure of contact and the length of time of the vibrating action at each spot should not cause soil to escape from beneath the edges of the tamping foot, thus tending to loosen the layer. Make a sufficient number of coverages to produce maximum density, as evidenced by practically no visible motion of surface particles adjacent to the edges of the tamping foot.

6.5.2.2 *Compaction by Sliding Weight Tamper*—Compact each layer of soil thoroughly by tamping blows uniformly distributed over the surface of the layer. Adjust the height of drop and give sufficient coverages to produce maximum density, depending on the coarseness and gravel content of the soil.

6.5.2.3 *Compaction by Other Methods*—Compaction may be accomplished by other approved methods, such as by vibratory packer equipment, where care is taken to obtain a uni-

form specimen without segregation of particle sizes (See Test Method D 2049).

6.5.3 *Relative Density Intermediate Between 0 and 100 %*—By trial in a separate container of the same diameter as the permeability cylinder, adjust the compaction to obtain reproducible values of relative density. Compact the soil in the permeability cylinder by these procedures in thin layers to a height about 2.0 cm (0.80 in.) above the upper manometer outlet.

NOTE 3—In order to bracket, systematically and representatively, the relative density conditions that may govern in natural deposits or in compacted embankments, a series of permeability tests should be made to bracket the range of field relative densities.

6.6 *Preparation of Specimen for Permeability Test*:

6.6.1 Level the upper surface of the soil by placing the upper porous plate or screen in position and by rotating it gently back and forth.

6.6.2 Measure and record: the final height of specimen, $H_1 - H_2$, by measuring the depth, H_2 , from the upper surface of the perforated top plate employed to measure H_1 to the top of the upper porous plate or screen at four symmetrically spaced points after compressing the spring lightly to seat the porous plate or screen during the measurements; the final weight of air-dried soil used in the test ($W_1 - W_2$) by weighing the remainder of soil, W_2 , left in the pan. Compute and record the unit weights, void ratio, and relative density of the test specimen.

6.6.3 With its gasket in place, press down the top plate against the spring and attach it securely to the top of the permeameter cylinder, making an air-tight seal. This satisfies the condition described in 3.1.1 of holding the initial density without significant volume change during the test.

6.6.4 Using a vacuum pump or suitable aspirator, evacuate the specimen under 50 cm (20 in.) Hg minimum for 15 min to remove air adhering to soil particles and from the voids. Follow the evacuation by a slow saturation of the specimen from the bottom upward (Fig. 2) under full vacuum in order to free any remaining air in the specimen. Continued saturation of the specimen can be maintained more adequately by the use of (1) deaired water, or (2) water maintained in an in-flow temperature sufficiently high to cause a decreasing temperature gradient in the specimen during the test. Native water or water

of low mineral content (Note 4) should be used for the test, but in any case the fluid should be described on the report form (Fig. 3). This satisfies the condition described in 3.1.2 for saturation of soil voids.

NOTE 4—Native water is the water occurring in the rock or soil *in situ*. It should be used if possible, but it (as well as de-aired water) may be a refinement not ordinarily feasible for large-scale production testing.

6.6.5 After the specimen has been saturated and the permeameter is full of water, close the bottom valve on the outlet tube (Fig. 2) and disconnect the vacuum. Care should be taken to ensure that the permeability flow system and the manometer system are free of air and are working satisfactorily. Fill the inlet tube with water from the constant-head tank by slightly opening the filter tank valve. Then connect the inlet tube to the top of the permeameter, open the inlet valve slightly and open the manometer outlet cocks slightly, to allow water to flow, thus freeing them of air. Connect the water manometer tubes to the manometer outlets and fill with water to remove the air. Close the inlet valve and open the outlet valve to allow the water in the manometer tubes to reach their stable water level under zero head.

7. Procedure

7.1 Open the inlet valve from the filter tank slightly for the first run to conditions described in 3.1.3, delay measurements of quantity of flow and heat until a stable head condition without appreciable drift in water manometer levels is attained. Measure and record the time, t , head, h (the difference in level in the manometers), quantity of flow, Q , and water temperature, T .

7.2 Repeat test runs at heads increasing by 0.5 cm in order to establish accurately the region of laminar flow with velocity, v , (where $v = Q/A$), directly proportional to hydraulic gradient, i (where $i = h/L$). When departures from the linear relation become apparent, indicating the initiation of turbulent flow conditions, 1-cm intervals of head may be used to carry the test run sufficiently along in the region of turbulent flow to define this region if it is significant for field conditions.

NOTE 5—Much lower values of hydraulic gradient, h/L , are required than generally recognized, in order to ensure laminar flow conditions. The following values are suggested: loose compactness ratings, h/L from 0.2 to 0.3, and dense compactness ratings, h/L from 0.3 to 0.5, the lower values of h/L applying to coarser soils and the higher values to finer soils.

7.3 At the completion of the permeability test, drain the specimen and inspect it to establish whether it was essentially homogeneous and isotropic in character. Any light and dark alternating horizontal streaks or layers are evidence of segregation of fines.

8. Calculations

8.1 Calculate the coefficient of permeability, k , as follows:

$$k = QL/At$$

where:

k = coefficient of permeability.
 Q = quantity of water discharged.
 L = distance between manometers.
 A = cross-sectional area of specimen.
 t = total time of discharge.
 h = difference in head on manometers.

8.2 Correct the permeability to that for 20°C (68°F) by multiplying k (see 8.1) by the ratio of the viscosity of water at test temperature to the viscosity of water at 20°C (68°F).

9. Report

9.1 The report of permeability test shall include the following information:

9.1.1 Project, dates, sample number, location, depth, and any other pertinent information.

9.1.2 Grain size analysis, classification, maximum particle size, and percentage of any oversize material not used.

9.1.3 Dry unit weight, void ratio, relative density as placed, and maximum and minimum densities.

9.1.4 A statement of any departures from these test conditions, so the results can be evaluated and used.

9.1.5 Complete test data, as indicated in the laboratory form for test data (see Fig. 3), and

9.1.6 Test curves plotting velocity, Q/A , versus hydraulic gradient, h/L , covering the ranges of soil identifications and of relative densities.

TABLE 1. Cylinder Diameter

Maximum Pore Size Line Between Sieve Opening	Maximum Cylinder Diameter			
	Less than 35 % of Total Soil Retained on Sieve Opening	More than 35 % of Total Soil Retained on Sieve Opening		
Sieve Opening	2.00-mm (No. 10)	9.5-mm (No. 10)	2.00-mm (No. 10)	9.5-mm (No. 10)
2.00-mm (No. 10)	76 mm (3 in.)	...	114 mm (4.5 in.)	...
9.5-mm (No. 10)	...	152 mm (6 in.)	...	229 mm (9 in.)
9.5-mm (No. 10) and 19.0-mm (No. 10)

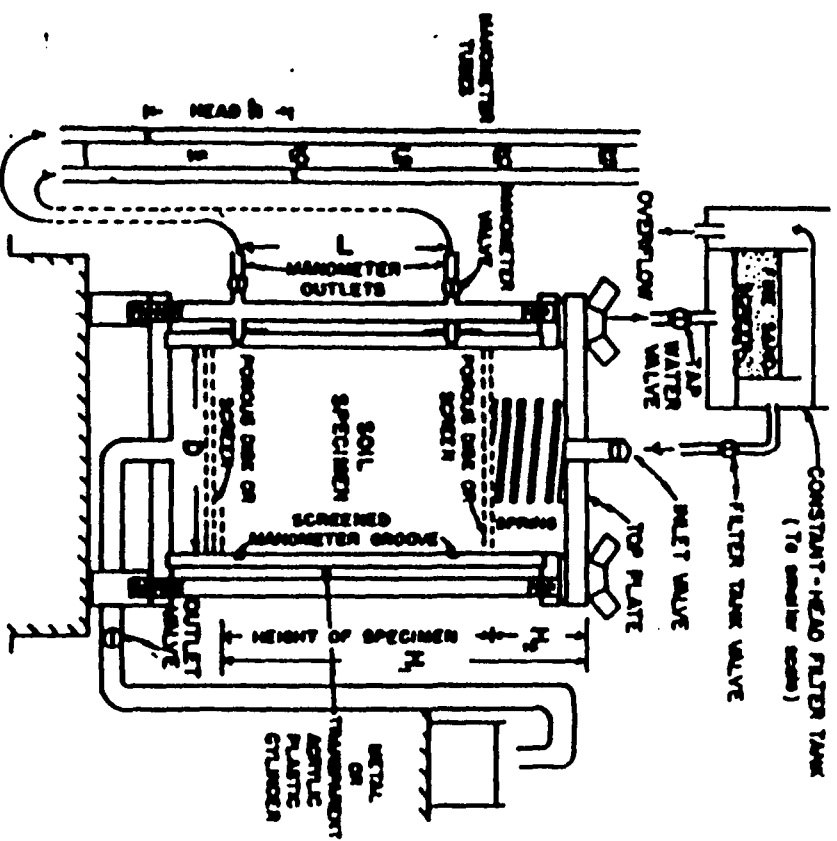


FIG. 1. Constant-Head Permeability

D 2434

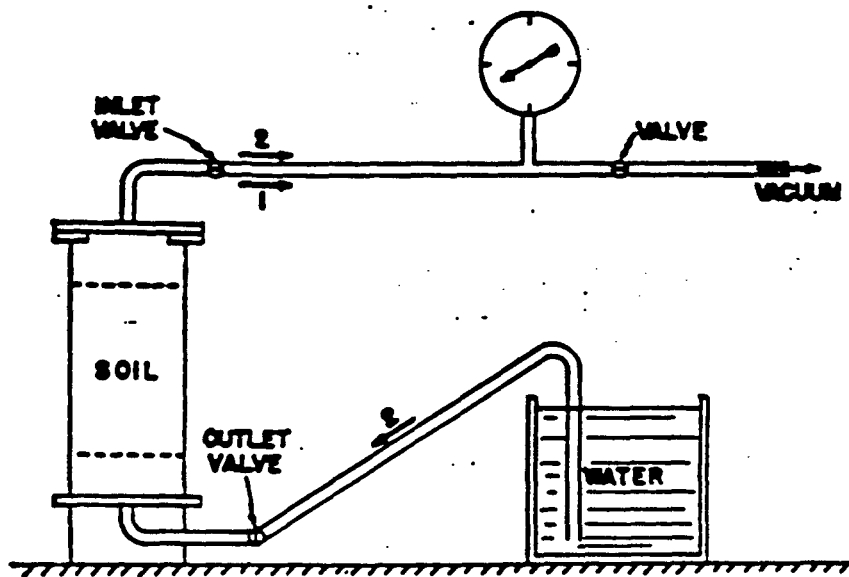


FIG. 2 Device for Evacuating and Saturating Specimen

D 2434

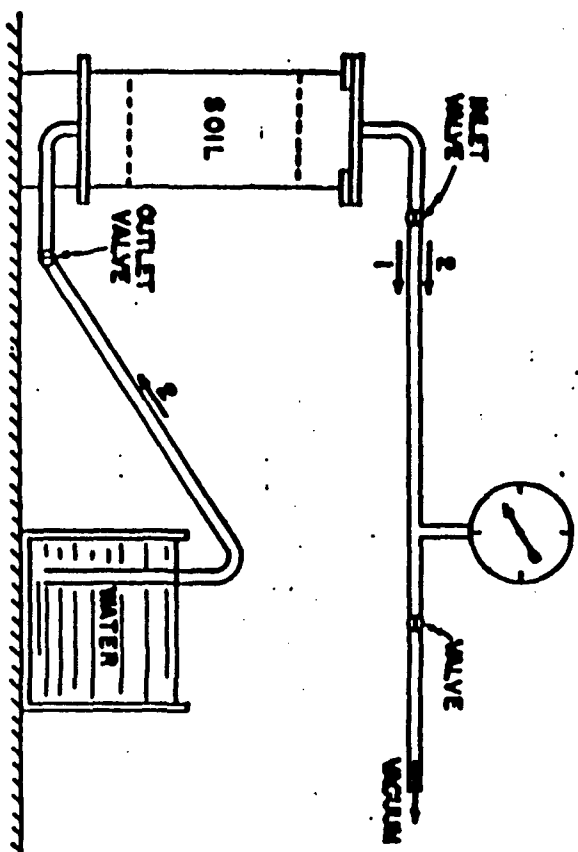


FIG. 2 Device for Evacuating and Saturating Specimens

D 2934

PERMEABILITY TEST ON GRANULAR SOIL

Test No. _____ Date of Test _____
 Location of Sample _____ Report _____
 Sample _____ Depth _____

(a) Description of Soil _____

Materials Used _____

(b) Unit Weight Determination
 Diameter, D , cm _____ Weight Balance, W_1 _____
 Area, A , cm² _____ Weight Afters, W_2 _____
 Length, L , cm _____ Weight Holes, W_3 _____
 Moisture Content (air-dried) _____
 Dry Unit Weight, $\Delta W^D W$ _____
 Void Ratio, e _____
 Relative Density, RD _____

(c) Permeability Test (Drops or Constant Head)

Test No.	Measurements		Head, h , cm	Q , cm ³	t , s	Q/A	h/L	Temperature, T , °C	k , cm/s
	H_1	H_2							
1									
2									
3									
4									
5									

FIG. 3 Permeability Test Data Sheet

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either approved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.

PERMEABILITY - NON GRANULAR SOILS

METHODS OF SOIL ANALYSIS

Part 1

Physical and Mineralogical Methods

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HYDRAULIC CONDUCTIVITY (PERMEABILITY) OF SATURATED SOILS

28-4.2 Falling-Head Method

28-4.2.1 PRINCIPLES

A diagram of the system used for measuring the hydraulic conductivity by the falling-head method is shown in Fig. 28-9. With a hydraulic head difference, H , across the sample, the volume of water dV that passes through the sample in time dt is given by

$$dV/dt = -K(H/L). \quad [9]$$

The differential volume of water, dV , may be replaced by adH , where a is the cross-sectional area of the standpipe. Integrating between limits t_1 , H_1 and t_2 , H_2 and solving for the conductivity yields the result:

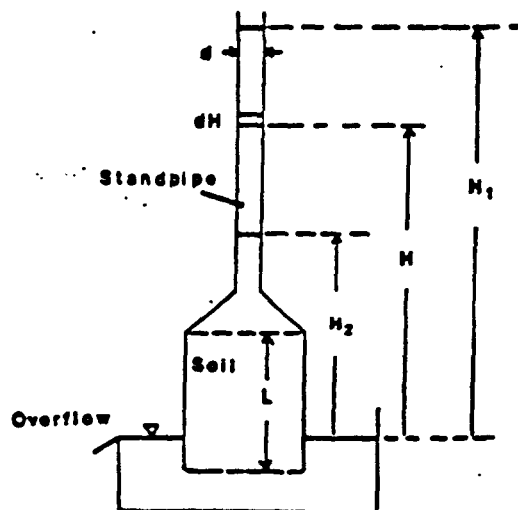


Fig. 28-9. Falling-head system for measuring hydraulic conductivity. L is the length of the soil sample, d is the diameter of the standpipe, dH is the change of hydraulic head that occurs in time dt , H is the hydraulic head difference across the sample at time t , H_1 is the initial and H_2 is the final hydraulic head difference.

$$K = (aL/At) \log_e (H_1/H_2) \quad [10]$$

where A is the cross-sectional area of the sample.

28-4.2.2 APPARATUS

The actual form of the apparatus may be quite varied. One possible arrangement is shown in Fig. 28-10. The support for the sample should have a high conductivity relative to that of the soil. A suitable screen, gauze, or cloth barrier may be fastened to the bottom of the sample cylinder, or a very high conductance porous stone such as those used in consolidation apparatus, may be used. The diameter of the standpipe should be chosen so that an easily measured change in head will occur in a reasonable time, say between 1 and 100 min. The required tube diameter may be estimated from

$$d = [K t D^2 / L (\log_e H_1)]^{1/2} \quad [11]$$

using appropriate values for the soil sample length, L , the diameter of the sample, D , the time, t , the hydraulic head ratio, $H_1 = H_1/H_2$, and the conductivity, K . With standpipe 2 cm in diameter and a soil sample with a diameter of 7.5 cm, a length of 5 cm, and a head ratio of 1.1, the largest conductivity that can be measured, using a fall time of 1 min, is

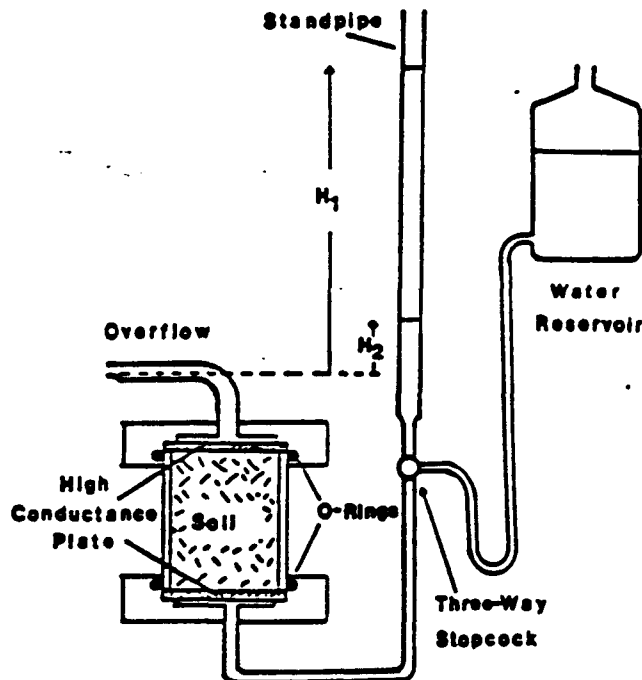


Fig. 28-10. Apparatus for measuring hydraulic conductivity by the falling-head method.

about 1×10^{-3} cm/s. The practical range of diameter for the standpipe is 0.2 to 2 cm. If the fall time is limited to 100 min or less and a standpipe with a diameter of 0.2 cm is used, the smallest K that can be measured is about 1×10^{-7} cm/s. This corresponds approximately to the lower limit of conductivity of silts and coarse clays.

28-4.2.3 PROCEDURE

The procedure given here is based on the apparatus shown in Fig. 28-10. If other falling-head apparatus is used, appropriate modifications in procedure will have to be made.

1. Install the end caps on the sample in its retaining cylinder, and wet the samples by supplying water to the bottom of the sample through the three-way stopcock and the lower porous plate. If complete rather than "natural" saturation of the sample is desired, the samples must be wetted under vacuum or by CO_2 -deaerated water flushing (see comments on method of wetting in section 28-4.1.5).
2. Fill the space above the sample up to the overflow with water. This may be done by flow upward through the sample, or by introduction of water with a pipet or syringe at the top of the sample. A 50-mL hypodermic syringe fitted with a large needle may conveniently be used for this purpose.
3. Establish a water level in the standpipe somewhat above the level chosen for H_1 , by introducing water through the three-way stopcock. The stopcock may be closed to hold the water in the standpipe until everything is ready to make the flow measurements.
4. Connect the standpipe to the sample by opening the stopcock, and measure the time for the water level to fall from H_1 to H_2 .
5. Additional measurements may be taken by repeating steps 3 and 4.
6. When the flow-rate measurements are finished, drain the excess water from the top of the sample and adjust the level of the water in the standpipe to the top of the sample.
7. Remove the end caps from the soil core, and transfer the soil into a moisture can. Determine the wet weight of the soil sample, W_w . Obtain the oven-dry weight of the sample, W_d .

28-4.2.4. CALCULATIONS

Calculate the hydraulic conductivity from Eq. [10].

Calculate the volumetric water content from

$$\theta = (W_w - W_d)/(d_s V_d).$$

Calculate the bulk density from

$$d_b = W_d/V_s.$$

28-4.2.5 COMMENTS

Most of the comments made in section 28-4.1.5 on the constant-head method of determining saturated hydraulic conductivity also apply to the falling head method.

The experimental arrangement shown in Fig. 28-8 may be used for either falling-head or constant-head measurements.

Conductivity measurements in relatively impervious materials ($K < 1 \times 10^{-7}$ cm/s) require excessively long measurement times with a standpipe of reasonable diameter. The conductivity of such materials may be measured by replacing the standpipe with a pressure transducer (Overman et al., 1968; Nightingale & Bianchi, 1970; Remy, 1973). The volume change per unit pressure change of many transducers is reasonably constant over a certain range of deformation of the diaphragm of the transducer. The volume change per unit pressure head change of the standpipe of a conventional falling-head system, i.e., its cross-sectional area, may be replaced by the reciprocal of the gauge sensitivity of the transducer, S , in the formula for calculating conductivity by the falling-head method

$$K = (L/AtS) \log_e (H_1/H_2) \quad [12]$$

Because the volume change per unit pressure change of many transducers, especially the diaphragm type, is generally smaller than that for a standpipe, the range of measurement of K may be extended to much lower values. However, certain precautions must be observed. Deformation of the apparatus and sample with changing pressure must be kept to a minimum. Unconfined samples of materials that have conductivities low enough that this method could be used will generally exhibit a change in consolidation with change in water pressure, thereby complicating the interpretation of the transducer response.

28-4.1.5 COMMENTS

The soaking procedure given above to wet the samples will not completely saturate them. Air will be trapped in the pores, and will tend to disappear slowly as deaerated water is passed through the sample. The degree of saturation obtained in the sample can be estimated by comparing the volumetric water content with the total porosity calculated from the bulk and particle densities.

The degree of saturation obtained by soaking may be representative of that obtained in situ when a soil is flooded with water, and may be called the *natural saturation*. The term *satiated* has also been used for this state of wetting. If the conductivity at total saturation is desired, a vacuum wetting procedure may be employed (see chapter 26 for details). The samples may also be wetted to complete saturation by flushing them with carbon dioxide, followed by wetting with deaerated water. After a few pore volumes of water have passed through the sample, the CO_2 will be dissolved in the water and the pore space will be filled with liquid. This procedure works well with coarse-textured samples. Precautions must be taken to prevent the introduction of air into the pores of the sample after flushing with CO_2 and before wetting with water. In some cases, the CO_2 deaerated water flushing procedure may be undesirable, because of the acidic solution that is formed when the deaerated water is introduced into the sample.

A wide range of conductivity values will be encountered in soils. Figure 28-6 shows the magnitudes of conductivity to be expected for a range of materials.

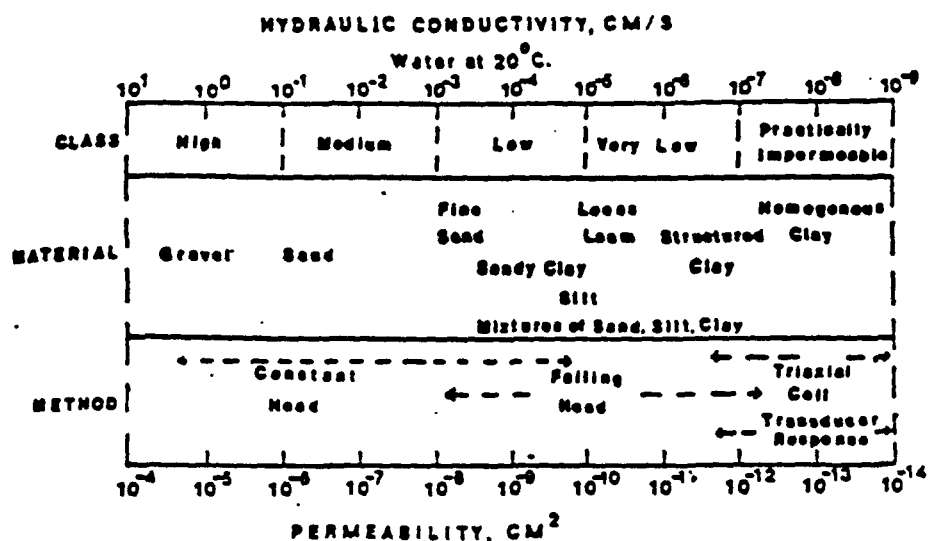


Fig. 28-6. Hydraulic conductivity of various materials at saturation.

The Darcy equation is not valid for all flow in porous media. Analysis of the various forces acting on the water passing through a porous medium shows that the Darcy equation should be valid when the inertial forces on the fluid are negligible compared to the viscous forces (Hubbert, 1957). As a practical matter, such a condition will prevail in silts and finer materials for any commonly occurring hydraulic gradient found in nature. In sands, especially the coarser sands, it will be necessary to restrict the hydraulic gradient to values less than about 0.5 to 1 to apply the Darcy equation. The range of validity of the Darcy equation can be demonstrated by measuring the flux density resulting from a series of applied gradients. The result should be a linear relation between the flux density and the hydraulic gradient. If the applied gradient is too large, the resulting flux density will be less than that predicted by the Darcy equation, using a conductivity calculated at low hydraulic gradients.

Swartzendruber (1969) discusses a number of reasons for deviations from the linear relation between flux density and gradient. Deviations have been observed, particularly in finer textured materials at low gradients. Some of these deviations have been traced to experimental problems, but in other cases no such explanation could be found. Non-Newtonian behavior of the fluid phase, changes in the soil matrix under flow, and electro-osmotic effects are some of the possible reasons for nonproportional behavior.

In many practical problems, nonproportional behavior is of little consequence, especially if the experimental error is large enough to mask the nonproportionality. If there is reason to doubt the applicability of the Darcy equation, tests should be made to determine its validity. Extremely large hydraulic gradients should be avoided, if possible, in con-

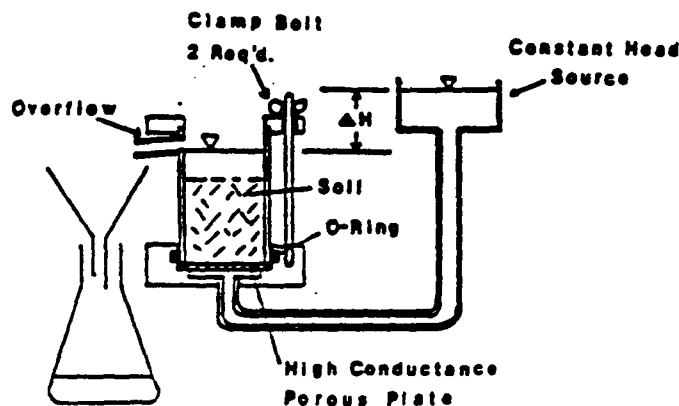


Fig. 28-7. Experimental arrangement for measuring hydraulic conductivity at low hydraulic gradient.

ductivity measurements if such gradients are unlikely to be encountered in the situations where the data are to be applied.

With the simple constant-head apparatus described above, the measurement error of the volumetric flow rate will become appreciable at 5 mL/h or less. If the sample has a diameter of 7.5 cm and the hydraulic gradient is about 1.5, this corresponds to a conductivity of approximately 2×10^{-3} cm/s. This indicates that a more sensitive method of measuring the volume flow rate is required for samples with low conductivity, or that another method must be used, such as the falling-head method described in section 28-4.2. The simple constant-head apparatus described above is not suitable for samples with very high conductivities, because the siphon tubes cannot deliver water fast enough to maintain a constant head of water on the sample. In such cases, an arrangement with hydraulic gradient less than unity, as shown in Fig. 28-7, may be used.

A more elaborate system for measuring conductivity may be warranted for studies of the physical principles of flow. The system for the steady-state measurement of the conductivity of unsaturated soil, described in section 28-5.1, may be used for the measurement of the conductivity of saturated soil. An alternative system is shown in Fig. 28-8. If a porous plate with a high conductance relative to that of the soil sample cannot be found for the lower end of the sample, it is better to install piezometers at two points along the axis of flow in the sample and calculate the hydraulic gradient for the sample section defined by the position of the tensiometers. The hydraulic head of the water in the piezometers may be measured with water manometers, but a more accurate measurement of the hydraulic gradient can be obtained with a differential pressure transducer connected between the two piezometers. The time required for the system to attain steady state is shorter for a transducer system than for a manometer system.

An alternative to the use of piezometers in the soil is to measure the head loss across the soil-plate system and correct for the conductance of the plate. The plate conductance, k_p , is defined by

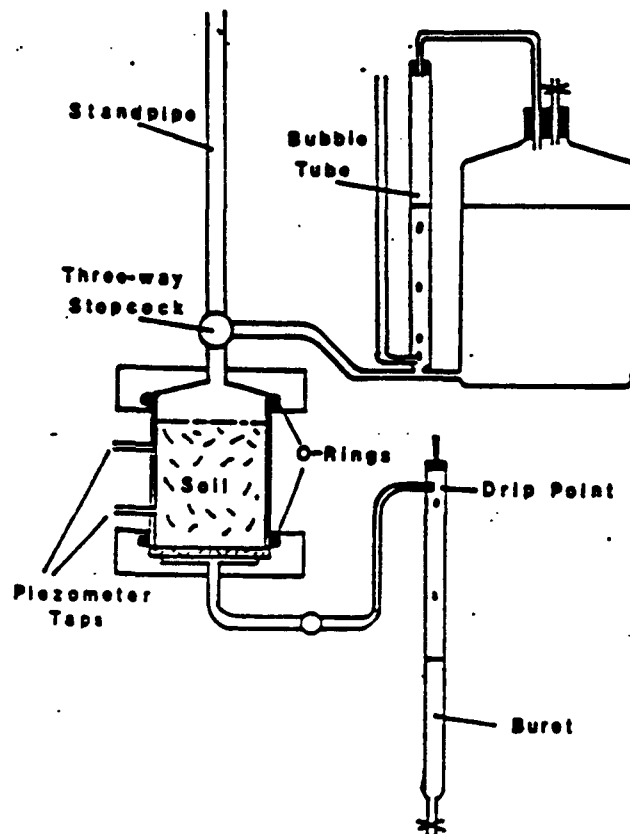


Fig. 28-8. A more elaborate apparatus for measuring hydraulic conductivity of saturated soils, which may be operated at constant head or falling head. The "side-arm", bubble tube minimizes aeration of the water supply.

$$k_p = (V/At)/\Delta H \quad [7]$$

where ΔH is the hydraulic head difference across the plate, and V is the volume of water passing through the plate of cross sectional area A in time t .

A separate measurement of the plate conductance is made. The conductivity of the soil-plate system, $\langle K \rangle$, is then measured, and the conductivity of the soil, K , is calculated from

$$K = L / (L_p/\langle K \rangle - 1/k_p) \quad [8]$$

where L_p is the thickness of the combined soil-plate system, and L is the thickness of the soil sample. In this procedure, it is assumed that the conductance of the plate does not change from the time that it is measured separately until it is used in the composite measurement, and that no significant contact resistance between the soil and the porous plate is present. The factor $1/k_p$, which is the resistance of the plate, should be less than about 10% of $L_p/\langle K \rangle$.

An appreciable error may be made in the measurement of low flow rates due to variations in the degree of "holdup" of water on the walls of a measuring buret. Evaporation losses also become relatively more significant. Precautions should be taken to minimize these errors when the flow rate is low. The movement of a meniscus in a *clean* horizontal calibrated tube may be used for low flow rate measurements with the flow cell shown in Fig. 28-8. The use of a short bubble (<1 cm) in a calibrated tube is subject to possible error due to water bypassing the bubble, and is not generally recommended.

TOC-soil

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request



Regional Transmittal



Telephone Request

- A. EPA Region/Client: II
- B. RSCC Representative: Jan Pels
- C. Telephone Number: (312) 353-2720
- D. Date of Request: _____
- E. Site Name: Hinco Dump, Elkhart Indiana

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

- General description of analytical service requested: Determination of organic carbon (%) in soil on air dried sample screened through a 100 or 140 mesh sieve. Applicable concentrations 0.1 - 2.0%, see Attachment 1
- Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):
20 low level soil samples
- Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):
Superfund remedial investigation

4. Estimated date(s) of collection: _____
5. Estimated date(s) and method of shipment: one shipment at end
of project by overnight carrier
6. Number of days analysis and data required after laboratory receipt of samples:
30
7. Analytical protocol required (attach copy if other than a protocol currently used in this program): —
See Attachment 7
8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): —
see Attachment 8
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.
see Attachment 9
10. Other (use additional sheets or attach supplementary information, as needed): _____
11. Name of sampling/shipping contact: Marcia Kuehl
Phone: (414) 458-8711

12. Data Requirements

Parameter	Detection Limit	Precision Desired (\pm % or Concentration)
% organic carbon	0.10%, report actual achieved if smaller	$\pm 20\%$ on lab duplicates

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
prep blank	1 in 10, max of 2	$\leq 0.1\%$
duplicate	1 in 5, max of 4	$\leq 20\%$ RPD or $< 0.2\%$ at concentrations of $0.1\% - 0.3\%$
positive control (lab determines)	1 in 10, max of 2	85-115%
instrument calibration checks + blanks	1 in 10	90-110% recovery for cal. check

14. Action Required if Limits are Exceeded

Take corrective action, reanalyze samples, contact SMO	$< 0.1\%$ total carbon for assumed routine sample weight

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

ATTACHMENT I

Determination of organic carbon(%) in soil, using sub-aliquots of air-dried soil, passed through a 100 mesh to 140 mesh screen. All of the sub-aliquot must pass the screen. Applicable organic carbon concentration range of interest is 0.1% to 2% (or larger) in soil, (dry weight basis). Laboratory may report lower concentration values.

Test procedures used for determining soil shall be the 1) Dry combustion (resistance furnace), 2) Dry combustion (induction furnace), 3) Dry combustion (automated methods), or 4) Wet combustion (combustion train) methods of analysis specified by Table 29-1 of "Methods of Soil Analyses," Part 2 - Chemical and Microbiological properties, 2nd ed., 1982, American Society of Agronomy, and Soil Science Society of America, Madison, Wisconsin. Copies of this copyrighted material are not being provided, because no laboratory doing organic carbon analysis of soil should be without it.

Any automated dry combustion test procedure used must provide results consistent with the other 3 methodologies and must be consistent with the requirements of Chapter 29, Sections 29-1, 29-2, and 29-3, "Methods of Soil Analysis" (MSA) Part II, 2nd ed., as appropriate. Soils can be calcareous or noncalcareous soils, with varying amounts of organic carbon. Soils determined may be subsurface as well as surface soils. If peat or muck soils are ever encountered, the laboratory will provide with the case narrative, limitations of any sample results and any solutions to problems encountered. This is also true for any other problem sample types encountered.

The laboratory, providing organic carbon analysis data, will provide information with the case narrative concerning methodology, instrumentation, and specific QA practices used for the set of soils tested. Requested information is detailed in items #8, and #9 of this SAS.

ATTACHMENT 7
Analytical Methods - Organic Carbon in Soil

- 7a. Sample Preparation: Representative sub-aliquot of air-dried soil (see % solids SAS) screened through 100 or 140 mesh as appropriate. All of the sub-aliquot must pass this screen.
- b. Test for Presence of Inorganic Carbon, MSA, Part II, Section 29-3.3.1. Place finely ground soil on a spot plate, and moisten with a few drops of water. Add 4 N HCl dropwise to the wetted sample and observe any effervescence. Allow sufficient time for dolomite to react (~5 min). If inorganic carbon is absent proceed with Total Carbon in items #7c, or 7d below. If inorganic carbon is present, or the test is not definitive, proceed with items #7e, or #7f prior to Total Carbon measurements of Item #7c or #7d.
- c. Total Carbon (Dry Combustion), MSA, Part II, Section 29-2.2.2. Use this as a guide for instrumental specifications. Instrument must test solid sample directly. Illustrative examples of this methodology are:
- 1) Total Carbon (Dry Combustion - Medium Temperature Resistance Furnace), MSA, Part II, Section 29-2.2.3.
 - 2) Total Carbon (Dry Combustion - High Temperature Induction Furnace), MSA, Part II, Section 29-2.2.4.
 - 3) Total Carbon (Dry Combustion - Other Instrumental Methods), MSA, Part II, Section 29-2.2.5. Any other instrumentation such as this must be justified and provide results as precise and accurate as the results from Sections 29-2.2.3, and 29-2.2.4.
- d. Total Carbon (Wet Digestion), MSA, part II, Section 29-2.3.2 Soil digested in 60:40 mixture of sulfuric acid and phosphoric acid (containing K_2CrO_7). CO_2 evolved is absorbed and weighed, or absorbed in standard base and titrated.
- 1) Specific examples are found in MSA, Part II, Figure 29-2, Figure 29-3, and Section 29-2.3.3.
- e. Pretreatment prior to Dry Combustion, MSA, Part II, Section 29-3.3.3. Inorganic carbon is removed by treating sample in a combustion boat, with 5% sulfurous acid (H_2SO_3). After several hours, remove the excess H_2SO_3 by leaving overnight in an evacuated dessicator. Read citation for further details.
- f. Pretreatment prior to Wet Digestion, MSA, Part II, Section 29-3.3.2. Inorganic carbon is removed by sulfuric acid - ferrous sulfate reagent in apparatus used for total carbon (Wet Digestion) prior to Total Carbon measurement. See citation for further details.

ATTACHMENT 7 (Cont.)

- g. Use only the methods specified above or obtain approval of CPMS, CRL prior to use of other method. Test procedure description, and description of specific measurement principles including equivalency to each of the 10 items of Figure 29-1 of MSA, part II and sample pretreatment of Section 29-3, MSA, Part II.
- h. Laboratory performing Total Carbon determinations must use and have a recognized procedure for removal of any inorganic carbon in sample.

ATTACHMENT 8

A variety of apparatus, instrumentation, sample preparation systems and read-outs can be used. It is the responsibility of the laboratory to provide appropriate QC audits and QC data with each set of samples tested.

If instrumentation requires calibration, provide calibration curve, including zero concentration standard and preparation blanks. Provide positive control (a test sample prepared independently from calibration standards) that provides a measure of accuracy of system. This should be done for all systems including gravimetric read-outs.

ATTACHMENT 9
Analytical Results Required

As part of Case Narrative, attach description of test procedure and instrumentation used for measurement of Total C and removal of any Inorganic C. Test procedure description must include sufficient information that the nature of specific analytical result deliverables can be determined including QC audits. In Case Narrative, discuss any problem type samples (including peat or muck soils), limitations on any sample results, and solution taken to resolve any problems. A sample preparation log will be provided, as appropriate.

Bench record tabulating any order of any sample weights and tare weights of absorbed CO₂, instrument calibrations, blanks, QA audits, etc., must be provided along with copies of any worksheets used to calculate results. Include copies of any instrument readouts. All must be legible. Report results as % organic Carbon on a dry weight basis (103-105°).

APPENDIX B

DQO SUMMARY SHEETS

DQO SUMMARY FORM

1. SITE NAME <u>Himco Dump</u> LOCATION <u>Elkhart, Indiana</u> NUMBER <u>IND 980500292</u>						EPA REGION <u>5</u> PHASE <u>I</u> (R1) R12 R13 ERA FS RD RA (CIRCLE ONE)																																						
2. MEDIA (CIRCLE ONE)		SOL	GW	SW/SD	AIR	SO	OTHER <u>FILL</u>																																					
3. USE (CIRCLE ALL THAT APPLY)		SITE CHARAC. (H&S)	RISK ASSESS.	EVAL ALTS.	ENGG DESIGN	PRP DETER.	MONITORING REMEDIAL ACTION	OTHER																																				
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CONTRACTOR <u>DONOHUE</u> PRIME CONTRACTOR _____ SITE MANAGER <u>VANESSA HARRIS</u> DATE <u>JAN' 1990</u>																																												

DQO SUMMARY FORM

1. SITE

NAME Himco Dump
LOCATION Elkhart, Indiana
NUMBER IND 980500292

EPA REGION 5
PHASE I

PI

PI 2

PI 3

ERA

FS

RD

RA

(CIRCLE ONE)

2. MEDIA

(CIRCLE ONE)

SOL

GW

SW/SED
WETLAND
SEDIMENT

AIR

BIO

OTHER

3. USE

(CIRCLE ALL THAT APPLY)

SITE CHARAC.
(H&S)

RISK ASSESS.

EVAL ALTS.

ENGG DESIGN

PPP DETER.

MONITORING REMEDIAL ACTION

OTHER

4. OBJECTIVE

DETERMINE IF CONTAMINANT SOURCE EXISTS AND IF RELEASES TO GROUNDWATER, SURFACE WATER, SEDIMENT OR AIR EXIST. - ECOLOGICAL RISK ASSESSMENT

5. SITE INFORMATION

AREA ~ 40 ACRES

DEPTH TO GROUND WATER 10-15 feet

GROUND WATER USE shallow aquifer wells abandoned, deep aquifer now used for

SOIL TYPES CaSO₄ as cap/fill, coarse sand & gravel, layer of silt clay drinking water

SENSITIVE RECEPTORS wetlands? residences immediately downgradient

6. DATA TYPES (CIRCLE APPROPRIATE DATA TYPES)

SW ONLY

A. ANALYTICAL DATA

PH

CONDUCTIVITY

VOL

ASB

TCLP

PESTICIDES

PCB

METALS

CYANIDE

D.O.

TOX

TOC

BTX

COO

TEMPERATURE

WATER QUALITY

B. PHYSICAL DATA

PERMEABILITY

POROSITY

GRAIN SIZE

BULK DENSITY

STAFF GAUGE

HYDRAULIC HEAD

PENETRATION TEST

HARDNESS

7. SAMPLING METHOD (CIRCLE METHOD(S) TO BE USED)

ENVIRONMENTAL

BIASED

GRAB

NON-INTRUSIVE

PHASED

SOURCE

GRID

COMPOSITE

INTRUSIVE

8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS)

LEVEL 1 FIELD SCREENING - EQUIPMENT PH COND DO METERS, THERMOMETER

LEVEL 2 FIELD ANALYSIS - EQUIPMENT

LEVEL 3 NON-CLP LABORATORY - METHODS

LEVEL 4 CLP/RAS - METHODS SW 2/88 7/88

LEVEL 5 NON STANDARD WATER QUALITY: CaSO₄ COD NH₃ NO₂ NO₃ TKN TP TDS TSS (SW ONLY) alkalinity bromide

9. SAMPLING PROCEDURES

BACKGROUND - 2 PER EVENT OR

CRITICAL (LIST) ALL

PROCEDURES SW: POND SAMPLER (DIP) SED: DREDGE WETLAND SED: HAND

DRIVEN SOIL PROBE

10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD)

A. FIELD

COLLOCATED - 5% OR

REPLICATE - 5% OR 10% (1)

FIELD BLANK - 5% OR 10% (1) (SW)

TRIP BLANK - 1 PER DAY OR 10% (1) (SW)

B. LABORATORY

REAGENT BLANK - 1 PER ANALYSIS BATCH OR PER SW/SAS

REPLICATE - 1 PER ANALYSIS BATCH OR

MATRIX SPIKE - 1 PER ANALYSIS BATCH OR

OTHER

11. BUDGET REQUIREMENTS

BUDGET

SCHEDULE DO SW/SED TOGETHER

STAFF 2 TECHS

CONTRACTOR DONOHUE

PRIME CONTRACTOR

SITE MANAGER VANESSA HARRIS

DATE JAN' 1990

DQO SUMMARY FORM

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1. SITE NAME <u>Himco Dump</u> LOCATION <u>Elkhart, Indiana</u> NUMBER <u>IND 980500292</u>		EPA REGION <u>5</u> PHASE <u>I</u> (R1) R2 R3 ERA FS RD RA (CIRCLE ONE)					
2. MEDIA (CIRCLE ONE)	SOL	GW EXISTING USES	SW/SED	AIR	BIO	OTHER	
3. USE (CIRCLE ALL THAT APPLY)	SITE CHARAC (H&S)	RISK ASSESS.	EVAL ALTS.	ENGG DESIGN	PRP DETER	MONITORING REMEDIAL ACTION	OTHER
4. OBJECTIVE <u>DETERMINE IF CONTAMINANT SOURCE EXISTS AND IF RELEASES TO GROUNDWATER, SURFACE WATER, SEDIMENT OR AIR EXIST. - DEVELOP GW TABLE MAP, ASSESS UTILITY OF PVC/SOLVENT GWED JOINT WELLS FOR CHEMISTRY MONITORING</u>							
5. SITE INFORMATION AREA <u>~ 40 ACRES</u> DEPTH TO GROUND WATER <u>10-15 feet</u> GROUND WATER USE <u>shallow aquifer wells abandoned, deep aquifer now used for</u> SOIL TYPES <u>CaSO₄ as cap/fill, coarse sand & gravel layer of silt clay drinking water</u> SENSITIVE RECEPTORS <u>Wetlands? residences immediately downgradient</u>							
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9. SAMPLING PROCEDURES BACKGROUND - 2 PER EVENT OR <u>5 UPGRADIENT WELLS</u> CRITICAL (LIST) <u>5 UPGRADIENT, M, E + P WELLS IMMEDIATELY DOWNGRADIENT</u> PROCEDURES <u>PURGE VIA VACK PUMP, SAMPLE WITH TEFLON BAILER</u>							
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DQO SUMMARY FORM

1. SITE NAME <u>Himco Dump</u> LOCATION <u>Elkhart, Indiana</u> NUMBER <u>IND 980500292</u>		EPA REGION <u>5</u> PHASE <u>I</u> RI1 <input checked="" type="radio"/> RI2 <input type="radio"/> RI3 <input type="radio"/> ERA <input type="radio"/> FS <input type="radio"/> RD <input type="radio"/> RA <input type="radio"/> (CIRCLE ONE)											
2. MEDIA (CIRCLE ONE)	SOL <input checked="" type="radio"/> <small>FROM MW PERMITS</small> GW <input type="radio"/> SW/SED <input type="radio"/> AIR <input type="radio"/> BIO <input type="radio"/> OTHER <input type="radio"/>												
3. USE (CIRCLE ALL THAT APPLY)	SITE CHARAC. (H&S) <input checked="" type="radio"/> RISK ASSESS. <input type="radio"/> EVAL. ALTS. <input checked="" type="radio"/> ENGG DESIGN <input type="radio"/> PPP DETER. <input type="radio"/> MONITORING REMEDIAL ACTION <input type="radio"/> OTHER <input type="radio"/>												
4. OBJECTIVE <u>DETERMINE IF CONTAMINANT SOURCE EXISTS AND IF RELEASES TO GROUNDWATER, SURFACE WATER, SEDIMENT OR AIR EXIST. - DETERMINE SURFACE GEOLOGY + SOIL CHEMISTRY</u>													
5. SITE INFORMATION AREA <u>~ 40 ACRES</u> DEPTH TO GROUND WATER <u>10-15 feet</u> GROUND WATER USE <u>shallow aquifer wells abandoned, deep aquifer now used for</u> SOIL TYPES <u>CaSO₄ as cap/fill, coarse sand + gravel, layer of silt clay drinking water</u> SENSITIVE RECEPTORS <u>wetlands? residences immediately downgradient</u>													
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9. SAMPLING PROCEDURES BACKGROUND - 2 PER EVENT OR <u>1 UPGRADIENT WELL LOCATION</u> CRITICAL (LIST) <u>UPGRADIENT WELL LOCATION</u> PROCEDURES <u>SPLIT SPOON, (DURING MW INSTALLATION)</u>													
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CONTRACTOR <u>DONOHUE</u> PRIME CONTRACTOR _____ SITE MANAGER <u>VANESSA HARRIS</u> DATE <u>JAN' 1990</u>													

DQO SUMMARY FORM

1. SITE		EPA REGION <u>5</u>	
NAME <u>Himco Dump</u>		PHASE <u>I</u>	
LOCATION <u>Elkhart, Indiana</u>		RI 1 <u> </u> RI 2 <u> </u> RI 3 <u> </u> ERA <u> </u> FS <u> </u> RD <u> </u> RA <u> </u>	
NUMBER <u>IND 980500292</u>		(CIRCLE ONE)	

2. MEDIA (CIRCLE ONE)	SOL	<u>GW</u> PRIVATE WELLS	SW/SED	AIR	BIO	OTHER
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3. USE (CIRCLE ALL THAT APPLY)	<u>SITE</u> CHARAC. (H&S)	<u>RISK</u> ASSESS.	<u>EVAL</u> ALTS.	ENGG DESIGN	PPP DETER	MONITORING REMEDIAL ACTION	OTHER
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4. OBJECTIVE DETERMINE IF CONTAMINANT SOURCE EXISTS AND IF RELEASES TO GROUNDWATER, SURFACE WATER, SEDIMENT OR AIR EXIST.

5. SITE INFORMATION

AREA ~ 40 ACRES DEPTH TO GROUND WATER 10-15 feet

GROUND WATER USE shallow aquifer wells abandoned, deep aquifer now used for

SOL TYPES CaSO4 as cap/fill, coarse sand & gravel, layer of silt clay drinking water

SENSITIVE RECEPTORS wetlands? residences immediately downgradient

6. DATA TYPES (CIRCLE APPROPRIATE DATA TYPES)

A. ANALYTICAL DATA

<p><u>PH</u></p> <p><u>CONDUCTIVITY</u></p> <p><u>VOA</u></p> <p><u>ASB</u></p> <p><u>TCLP</u></p>	<p><u>PESTICIDES</u></p> <p><u>PCB</u></p> <p><u>METALS</u></p> <p><u>CYANIDE</u></p> <p><u>DO</u></p>	<p><u>WATER QUALITY</u></p> <p><u>TOX</u></p> <p><u>TOC</u></p> <p><u>BTX</u></p> <p><u>COO</u></p> <p><u>TEMPERATURE</u></p>
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B. PHYSICAL DATA

<p><u>PERMEABILITY</u></p> <p><u>POROSITY</u></p> <p><u>GRAIN SIZE</u></p> <p><u>BULK DENSITY</u></p>	<p><u>HYDRAULIC HEAD</u></p> <p><u>PENETRATION TEST</u></p> <p><u>HARDNESS</u></p>
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7. SAMPLING METHOD (CIRCLE METHOD(S) TO BE USED)

ENVIRONMENTAL	BIASED	<u>GRAB</u>	NON-INTRUSIVE
SOURCE	GRID	COMPOSITE	INTRUSIVE

8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS)

LEVEL 1 FIELD SCREENING - EQUIPMENT PH, COND, DO METERS, HNU, THERMOMETER

LEVEL 2 FIELD ANALYSIS - EQUIPMENT

LEVEL 3 NON-CLP LABORATORY - METHODS

LEVEL 4 CLP/RAS - METHODS

LEVEL 5 NON STANDARD WATER QUALITY PARAMETERS, CRL / REGION 5 JAS FOR RESIDENTIAL WELLS
(SEE SW)

9. SAMPLING PROCEDURES

BACKGROUND - 2 PER EVENT OR

CRITICAL (LIST) ALL! (ATTEMPT TO SAMPLE 6 SHALLOW WELLS IF NOT PROPERLY ABANDONED)

PROCEDURES SAMPLE FROM TOP

10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD)

A. FIELD

COLLOCATED - 5% OR 10% (2)

REPLICATE - 5% OR 10% (2)

FIELD BLANK - 5% OR 10% (2)

TRIP BLANK - 1 PER DAY OR 10% (1)

B. LABORATORY

REAGENT BLANK - 1 PER ANALYSIS BATCH OR PER SAS

REPLICATE - 1 PER ANALYSIS BATCH OR

MATRIX SPIKE - 1 PER ANALYSIS BATCH OR

OTHER

11. BUDGET REQUIREMENTS

BUDGET

STAFF 2 TECHS

SCHEDULE

CONTRACTOR DONOHUE PRIME CONTRACTOR

SITE MANAGER VANESSA HARRIS DATE JAN' 1990

DDO SUMMARY FORM

1. SITE NAME <u>Himco Dump</u> LOCATION <u>Elkhart, Indiana</u> NUMBER <u>IND 980500292</u>		EPA REGION <u>5</u> PHASE <u>I</u> RI 1 <input checked="" type="radio"/> RI 2 <input type="radio"/> RI 3 <input type="radio"/> ERA <input type="radio"/> FS <input type="radio"/> RD <input type="radio"/> RA <input type="radio"/> (CIRCLE ONE)																		
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8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS) LEVEL 1 FIELD SCREENING - EQUIPMENT <u>PH, COND METERS, HNU, D.O. METER, THERMOMETER</u> LEVEL 2 FIELD ANALYSIS - EQUIPMENT _____ LEVEL 3 NON-CLP LABORATORY - METHODS _____ LEVEL 4 CLP/RAS - METHODS <u>SOW-2/88 7/88</u> LEVEL 5 NON STANDARD <u>WATER QUALITY PARAMETERS (SEE SW)</u>																				
9. SAMPLING PROCEDURES BACKGROUND - 2 PER EVENT OR _____ CRITICAL (LIST) <u>ALL</u> PROCEDURES <u>IF ACTIVE SEEP SEEN, DIG SUMP TO COLLECT</u>																				
10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD) <table style="width: 100%;"> <tr> <td colspan="2">A. FIELD</td> <td colspan="2">B. LABORATORY</td> </tr> <tr> <td>COLLOCATED - 5% OR _____</td> <td>REAGENT BLANK - 1 PER ANALYSIS BATCH OR <u>per SOW/SAS</u></td> <td>REPLICATE - 5% OR <u>10% (1)</u></td> <td>REPLICATE - 1 PER ANALYSIS BATCH OR _____</td> </tr> <tr> <td>FIELD BLANK - 5% OR <u>10% (1)</u></td> <td>MATRIX SPIKE - 1 PER ANALYSIS BATCH OR <u>✓</u></td> <td>TRIP BLANK - 1 PER DAY OR <u>10% (1)</u></td> <td>OTHER _____</td> </tr> </table>							A. FIELD		B. LABORATORY		COLLOCATED - 5% OR _____	REAGENT BLANK - 1 PER ANALYSIS BATCH OR <u>per SOW/SAS</u>	REPLICATE - 5% OR <u>10% (1)</u>	REPLICATE - 1 PER ANALYSIS BATCH OR _____	FIELD BLANK - 5% OR <u>10% (1)</u>	MATRIX SPIKE - 1 PER ANALYSIS BATCH OR <u>✓</u>	TRIP BLANK - 1 PER DAY OR <u>10% (1)</u>	OTHER _____		
A. FIELD		B. LABORATORY																		
COLLOCATED - 5% OR _____	REAGENT BLANK - 1 PER ANALYSIS BATCH OR <u>per SOW/SAS</u>	REPLICATE - 5% OR <u>10% (1)</u>	REPLICATE - 1 PER ANALYSIS BATCH OR _____																	
FIELD BLANK - 5% OR <u>10% (1)</u>	MATRIX SPIKE - 1 PER ANALYSIS BATCH OR <u>✓</u>	TRIP BLANK - 1 PER DAY OR <u>10% (1)</u>	OTHER _____																	
11. BUDGET REQUIREMENTS BUDGET _____ SCHEDULE _____ STAFF <u>2 TECHS</u>																				
CONTRACTOR <u>DONOHUE</u> PRIME CONTRACTOR _____ SITE MANAGER <u>VANESSA HARRIS</u> DATE <u>JAN' 1990</u>																				

DQO SUMMARY FORM

1. SITE NAME <u>Himco Dump</u> LOCATION <u>Elkhart, Indiana</u> NUMBER <u>IND 980500292</u>		EPA REGION <u>5</u> PHASE <u>I</u> RI 1 <input checked="" type="radio"/> RI 2 <input type="radio"/> RI 3 <input type="radio"/> ERA <input type="radio"/> FS <input type="radio"/> RD <input type="radio"/> RA <input type="radio"/> (CIRCLE ONE)					
2. MEDIA (CIRCLE ONE)	<input type="radio"/> SOL	<input type="radio"/> GW	<input type="radio"/> SW/SED	<input checked="" type="radio"/> AIR	<input type="radio"/> BIO	<input type="radio"/> OTHER	
3. USE (CIRCLE ALL THAT APPLY)	<input checked="" type="radio"/> SITE CHARAC. (H&S)	<input type="radio"/> RISK ASSESS.	<input type="radio"/> EVAL. ALTS.	<input checked="" type="radio"/> ENGG. DESIGN	<input type="radio"/> PRP DETER.	<input type="radio"/> MONITORING REMEDIAL ACTION	<input type="radio"/> OTHER
4. OBJECTIVE <u>DETERMINE IF CONTAMINANT SOURCE EXISTS AND IF RELEASES TO GROUNDWATER, SURFACE WATER, SEDIMENT OR AIR EXIST. ASSESS HOMEOWNERS BASEMENTS FOR CH₄, H₂S, EMANATING FROM LANDFILL</u>							
5. SITE INFORMATION AREA <u>~ 40 ACRES</u> DEPTH TO GROUND WATER <u>10-15 feet</u> GROUND WATER USE <u>shallow aquifer wells abandoned, deep aquifer now used for</u> SOIL TYPES <u>CaSO₄ as cap/fill, coarse sand & gravel, layer of silt clay drinking water</u> SENSITIVE RECEPTORS <u>wetlands? residences immediately downgradient</u>							
6. DATA TYPES (CIRCLE APPROPRIATE DATA TYPES)							
A. ANALYTICAL DATA				B. PHYSICAL DATA			
pH CONDUCTIVITY <input checked="" type="radio"/> VOA ASH TCLP	PESTICIDES PCB METALS CYANIDE <input checked="" type="radio"/> METHANE	TOX TOC BTX COO <input checked="" type="radio"/> HYDROGEN SULFIDE	PERMEABILITY POROSITY GRAIN SIZE BULK DENSITY	HYDRAULIC HEAD PENETRATION TEST HARDNESS			
7. SAMPLING METHOD (CIRCLE METHOD(S) TO BE USED)							
ENVIRONMENTAL	<input checked="" type="radio"/> BIASED	GRAB	NON-INTRUSIVE		PHASED		
SOURCE	GRID	COMPOSITE	<input checked="" type="radio"/> INTRUSIVE				
8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS)							
LEVEL 1 FIELD SCREENING - EQUIPMENT <u>VOAS BY HNU, CH₄/H₂S by CONTINUOUS GAS MONITOR</u>							
LEVEL 2 FIELD ANALYSIS - EQUIPMENT _____							
LEVEL 3 NON-CLP LABORATORY - METHODS _____							
LEVEL 4 CLP/RAS - METHODS _____							
LEVEL 5 NON STANDARD <u>VOAS ON SOLVENT TUBES</u>							
9. SAMPLING PROCEDURES							
BACKGROUND - 2 PER EVENT OR <u>1 OFFSITE UPWIND LOCATION</u>							
CRITICAL (LIST) <u>ALL - ESPECIALLY 6 HOMEOWNER BASEMENT SAMPLES</u>							
PROCEDURES <u>PERFORATED SOIL PROBE DRIVEN AT LEAST 3' INTO WASTE TENDRY - TENDRY/CHARGUAK TUBES</u> <u>IN TANDUM ATTACHED TO PERSONAL SAMPLING PUMP 1000ml/min & 10 ml</u>							
10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD)							
A. FIELD				B. LABORATORY			
COLLOCATED - 5% OR <u>10% (1)</u>				REAGENT BLANK - 1 PER ANALYSIS BATCH OR <u>PER SAS: DAILY</u>			
REPLICATE - 5% OR _____				REPLICATE - 1 PER ANALYSIS BATCH OR <u>10%</u>			
FIELD BLANK - 5% OR <u>10% - UNEXPOSED TUBES</u>				MATRIX SPIKE - 1 PER ANALYSIS BATCH OR <u>10%</u>			
TRIP BLANK - 1 PER DAY OR _____				OTHER _____			
11. BUDGET REQUIREMENTS							
BUDGET _____		SCHEDULE <u>DONE WITH WASTE/FILL SAMPLING FOR CHEMISTRY</u>					
STAFF <u>GEOLOGIST + TECH</u>							
CONTRACTOR <u>DONOHUE</u>		PRIME CONTRACTOR _____					
SITE MANAGER <u>VANESSA HARRIS</u>		DATE <u>JAN' 1990</u>					

DQO SUMMARY FORM

1. SITE NAME <u>Himco Dump</u> LOCATION <u>Elkhart, Indiana</u> NUMBER <u>IND 980500292</u>		EPA REGION <u>5</u> PHASE <u>I</u> (R1) (R2) (R3) (R4) (R5) (R6) (R7) (R8) (R9) (R10) (CIRCLE ONE)					
2. MEDIA (CIRCLE ONE)		SOL	GW	SW/SED	AIR	BIO	OTHER <u>WASTE-FILL</u>
3. USE (CIRCLE ALL THAT APPLY)		SITE CHARAC. (H&S)	RISK ASSESS.	EVAL ALTS.	ENGG DESIGN	PRP DETER.	MONITORING REMEDIAL ACTION
4. OBJECTIVE <u>DETERMINE IF CONTAMINANT SOURCE EXISTS AND IF RELEASES TO GROUNDWATER, SURFACE WATER, SEDIMENT OR AIR EXIST - PREDICT AMOUNT + RATE OF SETTLEMENT UNDER LOADS, DETERMINE STRENGTH OF WASTE + MATERIAL PROPERTIES</u>							
5. SITE INFORMATION AREA <u>~ 40 ACRES</u> DEPTH TO GROUNDWATER <u>10-15 feet</u> GROUND WATER USE <u>shallow aquifer wells abandoned, deep aquifer now used for</u> SOIL TYPES <u>CaSO₄ as cap/fill, coarse sand + gravel, layer of silty clay drinking water</u> SENSITIVE RECEPTORS <u>Wetlands? residences immediately downgradient</u>							
6. DATA TYPES (CIRCLE APPROPRIATE DATA TYPES) <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> A. ANALYTICAL DATA <div style="display: flex; justify-content: space-between;"> <div style="width: 30%;"> pH CONDUCTIVITY (VOA) (ABN) TCLP </div> <div style="width: 30%;"> PESTICIDES (PCB) METALS CYANIDE </div> <div style="width: 30%;"> TOX TOC BTX COD </div> </div> </div> <div style="width: 45%;"> B. PHYSICAL DATA <div style="display: flex; justify-content: space-between;"> <div style="width: 30%;"> PERMEABILITY POROSITY (GRAIN SIZE) BULK DENSITY (ATTENBERG LIMITS) </div> <div style="width: 30%;"> HYDRAULIC HEAD PENETRATION TEST HARDNESS (CONSOLIDATION) TRIAxIAL SHEAR </div> </div> </div> </div>							
7. SAMPLING METHOD (CIRCLE METHOD(S) TO BE USED) <div style="display: flex; justify-content: space-between;"> <div style="width: 30%;"> ENVIRONMENTAL SOURCE </div> <div style="width: 30%;"> BIASED (GRID) (250' centers) </div> <div style="width: 30%;"> GRAB COMPOSITE </div> <div style="width: 30%;"> NON-INTRUSIVE (INTRUSIVE) </div> </div>							
8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS) LEVEL 1 FIELD SCREENING - EQUIPMENT _____ LEVEL 2 FIELD ANALYSIS - EQUIPMENT _____ LEVEL 3 NON-CLP LABORATORY - METHODS _____ LEVEL 4 CLP/RAS - METHODS <u>SOW - 2/87, 7/88</u> LEVEL 5 NON STANDARD <u>GEOTECH / PHYSICAL</u>							
9. SAMPLING PROCEDURES BACKGROUND - 2 PER EVENT OR _____ CRITICAL (LIST) _____ PROCEDURES <u>0-6" FOR CHEMISTRY, 3' Shelby Tube FOR GEOTECH</u>							
10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD) <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> A. FIELD COLLOCATED - 5% OR <u>10% (3)</u> REPLICATE - 5% OR _____ FIELD BLANK - 5% OR _____ TRIP BLANK - 1 PER DAY OR _____ </div> <div style="width: 45%;"> B. LABORATORY REAGENT BLANK - 1 PER ANALYSIS BATCH OR <u>PER SOW/SAS</u> REPLICATE - 1 PER ANALYSIS BATCH OR _____ MATRIX SPIKE - 1 PER ANALYSIS BATCH OR _____ OTHER _____ </div> </div>							
11. BUDGET REQUIREMENTS BUDGET _____ SCHEDULE _____ STAFF <u>GEOLOGIST + TECH</u>							
CONTRACTOR <u>DONOHUE</u> PRIME CONTRACTOR _____ SITE MANAGER <u>VANESSA HARRIS</u> DATE <u>JAN' 1990</u>							

APPENDIX C

SAMPLE BOTTLE CLEANING PROTOCOLS

Table C-1

Bottle Cleaning Requirements
Himco Dump RI/FS
Elkhart, Indiana

<u>Container</u>	<u>Cleaning Protocol</u>	<u>QC Analyses</u>
120 ml wide mouth glass vials	A	TCL VOA
40 ml glass vials with Teflon septa		
80 oz amber glass bottles	B	TCL BNA and PCB/P
liter amber glass bottles		
8 oz clear glass jars	B	TAL metals
liter polyethylene bottles	C	TAL metals Cyanide
250 ml polyethylene bottles		

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Protocol A (volatile organics)

- wash glass vials, teflon-backed septa, teflon liners, and caps in hot tap water using laboratory grade non-phosphate detergent
- rinse three times with tap water
- rinse three times with ASTM Type 1 deionized water
- oven dry vials, septa, and liners at 125 degrees Celsius
- allow vials, septa, and liners to cool to room temperature in an enclosed contaminant-free environment
- seal vials with septa and cap
- each case of 72 tested for TCL volatile organics, test results included in case

Protocol B (extractable organics, metals)

- wash glass bottles, teflon liner, and caps in hot tap water laboratory grade non-phosphate detergent
- rinse three times with tap water
- rinse 1:1 nitric acid (metals-grade HNO_3 , ASTM deionized water)
- rinse three times with ASTM type 1 deionized water and dry
- rinse with pesticide grade methylene chloride or hexane
- oven dry at 125 degrees Celsius, allow to cool to room temperature in an enclosed contaminant-free environment
- place liners in lids and cap containers
- each case of 12 tested for TCL extractable organics, TAL metals, test results included in case

Protocol C (metals, cyanide)

- wash polyethylene bottles and caps in hot tap water with laboratory grade non-phosphate detergent
- rinse with 1:1 nitric acid (metals-grade HNO_3 , ASTM deionized water)
- invert and air dry in contaminant-free environment
- cap bottle
- each case of 12 tested for TAL metals and cyanide, test results included in case

Protocol C cleaned bottles will also be used for collection of BOD, COD, chloride, sulfate, TSS, TDS, alkalinity, TP, TKN, NH_3 , $\text{NO}_2 + \text{NO}_3$, and bromide. Testing of each case of 12 for these parameters will not occur. Bottle cleanliness will be assessed by filling one bottle of each lot with distilled water and submitting to the SAS laboratories with the field samples for analysis.

APPENDIX D

FIELD METER CALIBRATION PROCEDURES

ENU Model PI 101

Fisher Accumet Model 955

Cole Parmer Model 4070 Conductivity Meter

YSI Model 54A Dissolved Oxygen Meter

Mini CONRAD II Portable Radiological Survey Meter

Lumidor Gasponder IV Model PGM-14

HNu Model PI 101 Calibration

The calibration of the HNu is to be checked daily before field use by using a cylinder of isobutylene (HNu pn 101-350) with a regulator (HNu pn 101-351).

The ppm isobutylene reading, along with the span setting, is recorded in the calibration report contained in the HNu case (35 ppm, span 9.8).

In the field, the calibration must be checked daily before use and readjusted, if necessary, by using this cylinder and regulator as follows:

1. Connect the analyzer to the regulator and cylinder with a short piece of clean tygon tubing.
2. With the SPAN setting at 9.8 and the function switch at the same positions as listed on the Isobutylene Calibration Report, open the valve on the cylinder until a steady reading is obtained.
3. If the reading is 35 ppm, the analyzer calibration for the original species of interest is still correct.
4. If the reading has changed, adjust the SPAN setting until the reading is 35 ppm.
5. Shut off the cylinder as soon as the reading is established.
6. Record and maintain this new SPAN setting.

Notes:

- A. The regulator sets and controls the flow rate of gas at a value preset at the factory. This will be about 100 to 200 cc/min.; no adjustment to the regulator is necessary in the field.
- B. Do not use the cylinder below about 30 psig as readings below that level can deviate up to 10 percent from the rated value.
- C. Safely discard the disposable cylinder when empty and do not refill. It is against the law to transport refilled cylinders.

Fisher Accumet Model 955 Calibration

To measure the pH values of samples that vary over a range wider than two pH units, a two-point calibration will be done daily before field use to compensate for less than 100 percent electrode efficiency. The STANDARDIZE control provides the first set-point. The slope control sets the second set-point.

At pH 7, this "slope" adjustment has no effect on the readings. As readings increasingly differ from pH 7, the adjustment's effect becomes more pronounced - always in the opposite direction from one side of pH 7 to the other. Therefore, both calibration set points should be on the same side of pH 7.

Choose two buffer solutions with values that bracket the desired measuring range, usually pH 7 and pH 10 for groundwater and surface water. The buffer closest to pH 7 in value should always be used for the first set-point (with the STANDARDIZE control), and the buffer furthest from pH 7 in value should always be used for the second set-point (with the SLOPE control).

pH 7 Buffer Calibration:

1. Observe that electrode lead is securely connected to INPUT jack on instrument top panel. Also be sure to remove protective cot from tip of supplied combination electrode.
2. Observe that plug of ATC Probe is securely connected to °C jack on instrument right-side panel.
3. Obtain pH 7 buffer and ensure that buffer temperature is within 10°C of sample temperature.
4. Immerse electrode system and ATC Probe into buffer solution.
5. Set FUNCTION selector to °C position and place ON-OFF switch to ON position. Set the slope control full counter clockwise.
6. Allow electrode system and buffer solution to reach thermal equilibrium (°C reading steady), then determine exact pH of buffer solution from the table of buffer pH versus temperature found on the bottle label.
7. Set FUNCTION selector pH position, then adjust STANDARDIZE control until digital display indicates the pH value of buffer solution.
8. Remove electrode system and ATC Probe from buffer solution and rinse them with distilled water.

pH 10 (or 4) Buffer Calibration:

1. Immerse the electrode system and ATC probe into the pH 10 buffer.
2. Set the FUNCTION selector to C position. Allow the electrode system and buffer solution to reach thermal equilibrium (°C reading steady), then determine the exact pH of the buffer solution from the table of buffer pH versus temperature found on the bottle label.
3. Set the FUNCTION selector to the pH position, then adjust the SLOPE control until the digital display indicates the pH value of the buffer solution.
4. Remove the electrode system and ATC probe from the buffer solution and rinse with distilled water.
5. The pH meter is now ready for sample measurements.

Notes:

Thermal equilibrium normally requires about one minute, but will vary depending upon temperature difference between electrode system and buffers.

Cole Parmer Model 4070 Conductivity Meter

Two options for calibration of the Model 4070 are available in the field, depending on instrument accessory availability. If the pre-calibrated probe is available, this is the easiest calibration procedure to do in the field. However, if the probe is not available, calibration will have to be done daily before field use with a standard solution (commercially prepared):

Calibration with Pre-Calibrated Probe:

1. Connect a standard pre-calibrated probe to the unit.
2. Select the "Set K" position on the function switch.
3. Adjust the "Set K" control until the display indicates the value of the cell constant as marked on the probe being used.
4. The standard X 1.0 probe has a cell constant range from 0.80 to 1.20 and is calibrated to 2 decimal places. The display should be set to indicate this figure exactly.

The standard X 10 probe has a cell constant range from 8.0 to 12.0 and is calibrated to 1 decimal place. The display should be set to indicate this figure shifted one place to the right.

The standard X 0.1 probe has a cell constant range from 0.08 to 0.12 and is calibrated to 2 decimal places. The display should be set to indicate this figure shifted one place to the left.

5. Before using the 20 uS range, the probe should be thoroughly rinsed in deionized water, excess water shaken off and the outside of the probe body wiped dry. The display should then be set to zero, with the probe in free air, by using the "Zero 20 uS" control.

Calibration with a Standard Solution:

1. Select the "COND" range.
2. Immerse the conductivity cell and A.T.C. probe (if separate probe is being used) into the prepared standard.
3. Select the 2000 uS range if using a 1000 uS or 1413 uS standard or other suitable range if an alternative standard is being used.
4. Adjust the "Set K" control to give the exact readout of the standard solution selected on the display.
5. After carrying out the calibration with standard solutions as detailed above and with the probe still in the standard solution and the display set to the value of the standard solution, switch to the "Set K" range to give a direct readout of the cell constant.
6. If the readout cannot be set to the value of the standard solution with the "Set K" control, then the cell constant lies outside the "Set K" range of 0.80 to 1.20. In such cases, the display should be set to read 1.00 on the "Set K" position, the display reading on the range giving the best resolution should be noted and the cell constant calculated from the following formula:

K - Conductivity of Standard Solution
Display Reading

This figure should be noted and (with the cell constant set to 1.00 on the "Set K" position) the display reading should be multiplied by this figure to obtain the correct conductivity.

Note:

Ensure that the conductivity standard has not reached or gone over the expiration date marked on the container.

YSI Model 54A Dissolved Oxygen Meter Calibration

The air calibration procedure described below should be done daily before field use:

1. Place the probe in the YSI 5075A calibration chamber along with a few drops of distilled water.
2. Wait approximately 10 minutes for temperature stabilization.
3. Switch to TEMPERATURE and read. Refer to Table 1 - Solubility of Oxygen in Fresh Water and determine calibration value.
4. Determine altitude or atmospheric correction factor using Table II.
5. Multiply Table I calibration value by the Table II correction factor.
6. Switch to the appropriate mg/L range and adjust the CALIBRATE knob until the meter reads the value calculated in Step 5. Wait two minutes to verify calibration stability. Readjust if necessary.

Table 1 shows the amount of oxygen in mg/l that is dissolved in air saturated fresh water at sea level (760 mmHg atmospheric pressure) as temperature varies from 0° to 45°C.

TABLE 1
SOLUBILITY OF OXYGEN IN FRESH WATER

Temperature °C	mg/l Dissolved Oxygen	Temperature °C	mg/l Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derived from 15th Edition "Standard Methods for the Examination of Water and Wastewater."

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Table II shows the correction factor that should be used to correct the calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmospheric pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

TABLE II
CORRECTION FOR ATMOSPHERIC PRESSURE

Atmospheric Pressure <u>mmHg</u>	or	Equivalent Altitude <u>Ft.</u>	=	Correction <u>Factor</u>
775		540		1.02
760		0		1.00
745		542		.98
730		1094		.96
714		1688		.94
699		2274		.92
684		2864		.90
669		3466		.88
654		4082		.86
638		4756		.84
623		5403		.82
608		6065		.80
593		6744		.78
578		7440		.76
562		8204		.74
547		8939		.72
532		9694		.70
517		10472		.68
502		11273		.66

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

ARCS/P/HIMCO/AF1

MiniCONRAD II Portable Radiological Survey Meter

The manufacturer recommends that the Mini CONRAD only be calibrated semi-annually. Determine from the instrument log last date of calibration and assess if in-field calibration will be needed. The calibration procedure is as follows:

1. Place the range switch in the low (x1) position.
2. Remove the "x1" plastic screw that covers the low-range calibration control.
3. Turn the MiniCONRAD II on and place the target center in the appropriate field for the instrument being calibrated: Model 3034 3 mR/h.
4. Adjust the "x1" control for the proper indication on the meter scale.
5. Reduce the field by approximately 50 percent. The MiniCONRAD II must indicate within 15 percent of the field value.
6. Replace the "x1" plastic screw.
7. Place the range switch in the high (x100) position, remove the "x100" plastic screw and place the MiniCONRAD II target center in the appropriate field: Model 3034 300 mR/h.
8. Adjust the "x100" control for the proper indication on the meter scale. The field intensity is 100 times the value indicated.
9. Reduce the field by approximately 50 percent. The MiniCONRAD II reading, multiplied by 100, must be within 15 percent of the field.
10. Replace the "x100" plastic screw and turn the MiniCONRAD II OFF.

Note:

For calibration purposes, the "target center" is the intersection of the targets on the case.

Lumidor Gasponder IV Model PGM-14 Calibration

Calibration should be done at least daily before field use and after 12 hours of continuous use. Before proceeding, ensure that Gasponder is fully charged.

I. Initial Calibration

1. Instrument should be run at least 15 minutes.
2. Place Selector on "BATT.TEST" - check reading.
3. Place Selector on H_2S and set "ZERO."
4. Place Selector on CH_4 and set "ZERO."
5. Before proceeding with calibration, examine the calibration kit model PGM-14-10X to ensure that it contains:

One cylinder of Methane/CO	(Approx. 50 percent LEL/250 PPM CO)
One cylinder of H_2S	(Approx. 25 PPM)
One Gas Regulator and Hose	(Use only the regulator provided <u>with the</u> kit)

II. Calibration

1. Locate opening provided at side of main case to gain access to calibration adjustment for H_2S and CH_4 .
2. Attach flow regulator on Methane/CO cylinder.
3. With Selector set to " CH_4 ," check "ZERO" setting. If necessary, readjust with panel adjustment only.
4. Repeat Step 3 for " H_2S ."
5. Return Selector switch to the " CH_4 " position. Turn on flow regulator, and connect hose to unit using quick disconnect fitting.
6. After one minute, adjust calibration screw until reading is the same as stated on cylinder label (i.e., 50 percent LEL).
7. Turn off flow regulator and remove regulator from cylinder.
8. Attach flow regulator to cylinder marked H_2S (RED).
9. Turn selector switch to the H_2S position. Turn on flow regulator and connect hose to unit using quick disconnect fitting. After three minutes, adjust calibration screw until reading is the same as stated on cylinder (i.e., 25 PPM). Disregard " O_2 " alarm that occurs during this test.
10. When calibration procedure is completed, remove the gas regulator from cylinder, carefully repack all components and replace calibration cover securely.

ARCS/P/HIMCO/AE8

APPENDIX E
STANDARD OPERATING PROCEDURES
FOR
FIELD MEASUREMENTS

Volatile Organics by HNU

pH

Conductivity

Dissolved Oxygen

Radioactivity

Methane and Hydrogen Sulfide

STANDARD OPERATING PROCEDURE FOR THE FIELD MEASUREMENT OF VOLATILE ORGANICS BY HNu

I. PARAMETER(S)

Volatile organics with ionization potentials of less than 10.2 eV.

II. RANGE OF MEASUREMENT

The linear range is 0.1-400 ppm, the useful range is 0.1-2000 ppm.

III. LIMIT OF DETECTION

The detection limit of 0.1 ppm was determined by the manufacturer based on the response of benzene at a span setting of 9.8 and a 10.2 eV probe.

IV. SAMPLE MATRICES

Headspace above soil samples collected in split spoon samplers or with trowels.

V. PRINCIPLE, SCOPE AND APPLICATION

The analyzer measures the concentration of trace gases present in the atmosphere by photoionization. Photoionization occurs when an atom or molecule absorbs a photon of sufficient energy to release an electron and become a positive ion. This will occur when the ionization potential of the molecule in electron volts (eV) is less than the energy of the photon. The source of photons is an ultraviolet lamp with an energy of 10.2 eV.

Sample gases enter through the inlet into the ion chamber and are exposed to photons emanating from the ultraviolet lamp. Ionization occurs for those molecules having ionization potentials near to or less than that of the lamp. A positive-biased polarizing electrode causes these positive ions to travel to a collector electrode in the chamber. Thus the ions create an electrical current which is amplified and displayed on the meter. This is proportional to the concentration of trace gas present in the ion chamber and to the sensitivity of that gas to photoionization.

In service, the analyzer is first calibrated with a gas of known composition equal, close to, or representative of that to be measured. Gases with ionization potentials near to or less than 10.2 eV will be ionized. These gases will thus be detected and measured by the analyzer. Gases with ionization potentials higher than 10.2 eV will not be detected.

Ionization potentials for various atoms, molecules and compounds are given in the Instruction Manual Appendix. The ionization potential of the major components of air, i.e., oxygen, nitrogen, and carbon dioxide, range from about 12.0 eV to about 15.6 eV and are not ionized by any of the three lamps. Gases

with ionization potentials near to or slightly higher than the lamp are partially ionized, with low sensitivity.

VI. INTERFERENCES AND CORRECTIVE ACTIONS

Air currents or drafts in the vicinity of the probe tip may cause fluctuations in readings. Soil, dust, or debris drawn into the probe will result in low readings and/or negative deflection of the meter. High ambient humidity and high percent moisture samples will cause negative deflection of the meter.

To obtain stable, reproducible readings, corrective actions such as shielding the probe from drafts or currents and rain should be done. Sample moisture content is not controllable as representativeness dictates that no special handling of the sample occurs that might bias chemical results. Any drying of the sample would result in loss of volatiles. The field records should indicate which samples were wet and note any negative meter deflection.

VII. SAFETY PRECAUTIONS

Do not look at the light source closer than 6 inches with unprotected eyes. Observe only if necessary, then only briefly. Continued exposure to ultraviolet energy generated by the light source can be harmful to eyesight.

The instrument measures gases in the vicinity of the operator and a high reading when measuring toxic or explosive gases should be cause for action for operator safety.

Use HNu only in an emergency with a low battery when on battery charge.

Turn the function switch on the control panel to the OFF position before any disassembly. Otherwise, high voltage of 1200 vdc will be present. Use great care when operating the analyzer with the readout assembly outside its case due to the presence of 1200 vdc.

Do not interchange lamps of different eV ratings in a probe. Amplifier and components are selected for a specific eV lamp. A probe with the wrong lamp will not operate properly.

VIII. SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING

Soil

Measurement is done on the soil contained in the split spoon sampler or trowel placed in a clean 8-ounce jar with a teflon-lined lid. Measurement should be made within 5 minutes of collection in the field. The jar should be half full.

IX. APPARATUS

- Clean 8-ounce clear glass jars with teflon-lined lid, hole the diameter of the HNu probe drilled in top.

- Duct tape.
- HNu Model PI 101.
- Isobutylene calibration gas cylinder and regulator.
- Spare 10.2 eV lamps.
- Battery charger.

X. ROUTINE PREVENTIVE MAINTENANCE

1. Battery

Check the battery charge during each period of operation, at least once daily. If the battery is low as indicated by the meter reading or the warning indicator, it is necessary to recharge the battery.

2. Lamp

During periods of operation of the analyzer, dust or other foreign matter could be drawn into the probe forming deposits on the surface of the UV lamp or in the ion chamber. This condition is indicated by meter readings that are low, erratic, unstable, non-repeatable, or drifting, or show apparent moisture sensitivity. These deposits interfere with the ionization process and cause erroneous readings. Check for this condition daily. Cleaning can be accomplished as follows:

- a. Disassemble the probe and remove the lamp and ion chamber. Exercise great care in doing so to prevent inadvertent damage to these components.
- b. First check the lamp window for fouling by looking at the surface at an incident angle. Any deposits, films or discoloration may interfere with the ionization process. Clean the window as follows:
 - 1) First clean by rubbing gently with lens tissue dipped in a detergent solution.
 - 2) If this does not remove deposit, apply a small amount of HNu cleaning compound (PA101534) directly onto the lens of the lamp and spread evenly over surface with a non-abrasive tissue (e.g. Kim-Wipe) or a lens tissue.
 - 3) Wipe off compound with a new tissue.
 - 4) Rinse with a warm water (about 80°F) or damp tissue to remove all traces of grit or oils and any static charge that may have built up on the lens. Dry with new tissue.
 - 5) Reinstall lamp in detector and check analyzer operation.
 - 6) If performance is still not satisfactory, replace the lamp.

3. Ion Chamber

- a. Inspect the ion chamber for dust or particulate deposits. If such matter is present, the chamber can be cleaned by removing the outer Teflon ring, and the four screws holding the retaining ring. Carefully move the retaining ring aside (NOTE: this is soldered) and remove the screen. A tissue or cotton swab, dry or wetted with methanol, can be used to clean off any stubborn deposits. The assembly can also be gently swirled in methanol and dried gently at 50-60°C for approximately a half hour. No liquid must be present at reassembly as this would affect the performance. Do not clean the ion chamber with the HNu cleaning compound.
- b. Reassemble the probe and check analyzer operation.
- c. If performance is still not satisfactory, replace the lamp.

XI. REAGENTS AND CALIBRATION STANDARDS

The calibration gas cylinder containing a certified value of isobutylene is the only reagent/standard. Replace when empty, no shelf life is applicable.

XII. CALIBRATION PROCEDURES

The calibration of the HNu is to be checked daily before field use by using a cylinder of isobutylene (HNu pn 101-350) with a regulator (HNu pn 101-351).

The ppm isobutylene reading, along with the span setting, is recorded in the calibration report contained in the HNu case (35 ppm, span 9.8).

In the field, the calibration must be checked daily before use and readjusted, if necessary, by using this cylinder and regulator as follows:

1. Connect the analyzer to the regulator and cylinder with a short piece of clean tygon tubing.
2. With the SPAN setting at 9.8 and the function switch at the same positions as listed on the Isobutylene Calibration Report, open the valve on the cylinder until a steady reading is obtained.
3. If the reading is 35 ppm, the analyzer calibration is still correct.
4. If the reading has changed, adjust the SPAN setting until the reading is 35 ppm.
5. Shut off the cylinder as soon as the reading is established.
6. Record and maintain this new SPAN setting.

Notes:

- A. The regulator sets and controls the flow rate of gas at a value preset at the factory. This will be about 100 to 200 cc/min.; no adjustment to the regulator is necessary in the field.
- B. Do not use the cylinder below about 30 psig as readings below that level can deviate up to 10 percent from the rated value.

- C. Safely discard the disposable cylinder when empty and do not refill. It is against the law to transport refilled cylinders.

XIII. SAMPLE PREPARATION

A. Soil

Soil obtained in split spoon samplers or by a hand trowel/corer should be placed in a clean 8-ounce clear glass jar outfitted with a teflon-lined lid with a hole the diameter of the HNu probe drilled in it. Fill the jar half full, close tightly (lid hold should be covered with duct tape) and allow to equilibrate for 2 minutes before measurement.

XIV. ANALYTICAL MEASUREMENT

1. Unclamp the cover from the main readout assembly.
2. Remove the inner lid from the cover by pulling out the two fasteners.
3. Remove the probe, handle and cable from the cover. Attach the handle to the front part of the probe.
4. Connect the probe cable plug to the 12 pin keyed socket on the readout assembly panel. Carefully match the alignment slot in the plug to the key in the connector. Screw down the probe connector until a distinct snap and lock is felt.
5. Screw the probe extension into the probe end cap. The probe may be used without the extension if desired.
6. Set the SPAN control for 10.2 eV as specified by the daily in-field calibration with isobutylene.
7. Turn the function switch to the BATT (battery check) position. The needle on the meter will go to the green zone if the battery is fully charged. If the needle is below the green arc or if the Low Battery Indicator comes on, the battery must be recharged before the analyzer is used.
8. Set SPAN pot to 9.8.
9. Turn the function switch to the STANDBY position. Turn the zero adjustment until the meter needle is at zero.
10. Calibrate the instrument daily as described in Section 12.
11. Turn the function switch to the appropriate operating position. Start with the 0-2000 position and then switch to the more sensitive ranges. The UV light source should be on, confirmed by briefly looking into the probe to observe the purple glow from the lamp.

12. Peel back the duct tape from the lid and place the HNu probe in the hole. Record the reading on the Soils Data Form.
13. After completion of each days measurements, check battery condition as described in No. 7.
14. Turn function switch to OFF position.
15. When not operating, leave analyzer in assembled condition, and connected to battery charger.
16. When transporting, disassemble probe and extension from readout assembly and return equipment to its stored condition.
17. In case of emergency, turn function switch to OFF position.

XV. FLOW CHART

Not applicable.

XVI. DATA TREATMENT

Not applicable, instrument provides direct real time readout in ppm units.

XVII. DATA DELIVERABLES

Record calibration details on Field Instrument Calibration Log and results on Soils Data Form.

XVIII. QUALITY CONTROL REQUIREMENTS

Each day an upwind location will be used to determine the ambient background level. One location for every ten will be selected for measurement as a field duplicate. Two separate aliquots of soil will be placed in two 8-ounce jars. No rpd limits have been established for field duplicate precision. Record both reading on the Soils Data Form.

XIX. REFERENCES

- Instruction Manual, HNu Model PI 101 Portable Ionization Analyzer, December, 1985.
- HNu Systems (617) 964-6690

XX. METHOD VALIDATION DATA

Not applicable as volatile organics are being measured for field screening purposes to select sampling locations for chemical analysis.

ARCS/PROJ/HIMCO/AF3

**Standard Operating Procedure
for the
Measurement of pH in the Field
Using the Fisher Accumet Model 955 Portable
pH/mV Temperature Meter**

1. Parameter to be measured: pH.
2. Range of Measurement: 0.00 to 14.00 pH units.
3. Sample Matrix.

This method is applicable to drinking, surface, saline, and groundwater samples; chemical domestic and industrial wastes; leachates.

4. Principle, Scope, and Application.

The pH of a sample is determined electrometrically, using a combination sample-reference pH electrode.

5. Interferences and Corrective Action.

- 5.1 Any sample constituent which coats the electrode can cause sluggish response. This can be eliminated by cleaning the electrode according to manufacturer's instructions.
- 5.2 Temperature effects on the electrometric measurement of pH arise from two sources. The first source is caused by change in electrode output at various temperatures. This is avoided by using the Automatic Temperature Compensation (ATC) probe. The second source is the change of pH inherent in the sample at various temperatures. Therefore, the sample temperature should be reported with the pH.

6. Safety Precautions.

Common sense will eliminate the possibility of any hazards while the portable pH meter is being used. Obviously, buffers and field samples must never be pipetted by mouth. Handle the electrode carefully to avoid breakage. Since the electrode will be measuring potentially hazardous material and all pH electrodes leak small quantities of electrolytes, they must never be placed in the mouth.

7. Sample Size, Collection, Preservation, and Handling.

Collect approximately 50 ml or more of the sample to be measured. To avoid the possibility of cross contamination from sample to sample and contamination of leaking electrolyte, the samples will be discarded after pH measurement and not used for any other parameters. If for any reason the pH cannot be taken immediately in the field, samples must be stored at 4°C and measured within a 24-hour period.

8. Apparatus and Materials.

- 8.1 Fisher Accumet Model 955 Portable pH/mV Temperature Meter
- 8.2 Three or more 50 ml plastic beakers
- 8.3 Commercially prepared pH 4.01, 7.00, 10.00 buffers
- 8.4 Squeeze type wash bottle, 125 ml or larger
- 8.5 Distilled water
- 8.6 Electrode; extra electrolyte for filling if necessary
- 8.7 Automatic Temperature Compensation (ATC) Probe
- 8.8 One or two 9-volt transistor batteries

9. Routine Preventive Maintenance.

- 9.1 The instrument and batteries should be checked and calibrated in the laboratory before the field effort begins.
- 9.2 The accuracy of the buffer solutions used for field and laboratory calibration should be checked. Buffer solutions need to be changed due to degradation upon exposure to the atmosphere. (The date of preparation of each buffer should be included on the bottle label.) Record source of buffer and date opened on Field Meter Log Sheet.
- 9.3 Immerse the tip of the electrodes in water overnight. If this is not possible due to field conditions, immerse the electrode tip in water for at least an hour before use. The electrode tip may be immersed in a rubber or plastic sack containing buffer solution for field transport or storage. This is not applicable for all electrodes, as some must be stored dry.
- 9.4 Make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
- 9.5 Distilled water pH and conductivity should be monitored periodically.
- 9.6 The electrode should be stored and cleaned according to manufacturer's instructions.

10. Reagents and Calibration Standards.

Commercially prepared standardized buffers of pH 4.01, 7.00, and 10.00 are used to calibrate the instrument. The source and date opened will be recorded on the Field Meter Log Sheet.

11. Calibration Procedure.

Fisher Accumet Model 955 Calibration

To measure the pH values of samples that vary over a range wider than two pH units, a two-point calibration will be done daily before field use to compensate for less than 100 percent electrode efficiency. The STANDARDIZE control provides the first set-point. The slope control sets the second set-point.

At pH 7, this "slope" adjustment has no effect on the readings. As readings increasingly differ from pH 7, the adjustment's effect becomes more pronounced - always in the opposite direction from one side of pH 7 to the other. Therefore, both calibration set points should be on the same side of pH 7.

Choose two buffer solutions with values that bracket the desired measuring range, usually pH 7 and pH 10 for groundwater and surface water. The buffer closest to pH 7 in value should always be used for the first set-point (with the STANDARDIZE control), and the buffer furthest from pH 7 in value should always be used for the second set-point (with the SLOPE control).

pH 7 Buffer Calibration:

- a. Observe that electrode lead is securely connected to INPUT jack on instrument top panel. Also be sure to remove protective cot from tip of supplied combination electrode.
- b. Observe that plug of ATC Probe is securely connected to °C jack on instrument right-side panel.
- c. Obtain pH 7 buffer and ensure that buffer temperature is within 10°C of sample temperature.
- d. Immerse electrode system and ATC Probe into buffer solution.
- e. Set FUNCTION selector to °C position and place ON-OFF switch to ON position. Set the slope control full counter clockwise.
- f. Allow electrode system and buffer solution to reach thermal equilibrium (°C reading steady), then determine exact pH of buffer solution from the table of buffer pH versus temperature found on the bottle label.
- g. Set FUNCTION selector pH position, then adjust STANDARDIZE control until digital display indicates the pH value of buffer solution.
- h. Remove electrode system and ATC Probe from buffer solution and rinse them with distilled water.

pH 10 (or 4) Buffer Calibration:

- a. Immerse the electrode system and ATC probe into the pH 10 buffer.
- b. Set the FUNCTION selector to C position. Allow the electrode system and buffer solution to reach thermal equilibrium (°C reading steady), then determine the exact pH of the buffer solution from the table of buffer pH versus temperature found on the bottle label.
- c. Set the FUNCTION selector to the pH position, then adjust the SLOPE control until the digital display indicates the pH value of the buffer solution.
- d. Remove the electrode system and ATC probe from the buffer solution and rinse with distilled water.
- e. The pH meter is now ready for sample measurements.

Notes:

Thermal equilibrium normally requires about 1 minute, but will vary depending upon temperature difference between electrode system and buffers.

12. Sample Preparation.

There is no sample preparation for this procedure other than allowing the electrode system and sample solution to reach thermal equilibrium (steady thermometer reading).

13. Analytical Measurement.

- 13.1 Calibrate the instrument (See Section 11).
- 13.2 Remove electrode system and ATC Probe from buffer solution and rinse them with distilled water.
- 13.3 Immerse the electrode system and ATC Probe into the sample solution and allow sufficient time for the electrode system and sample solution to reach thermal equilibrium.

Notes: Thermal equilibrium normally requires about 1 minute, but will vary depending upon temperature difference between electrode system and sample.

- 13.4 Read the pH value of sample from the digital display and record on the Well Purging and Sample Collection form. Remove electrode system and ATC probe from sample and rinse with distilled water. Repeat step 13.3 and 13.4 for remaining samples.
- 13.5 ON-OFF switch to OFF after last measurement.

14. Data Deliverables.

The pH accuracy will be assessed by performing two measurements on three standard buffer solutions. Each measurement will be within $\pm 0.05\%$ standard unit of the certified value for the buffer solutions. Precision will be assessed through replicate measurements on field samples. The standard deviation of four replicate measurements must be less than or equal to 0.1 standard unit. The electrode will be withdrawn, rinsed with deionized water, and re-immersed between each replicate. Calibration and verification will be done in the field before the first replicate and after the last. The instrument used will be capable of providing measurements of 0.01 standard unit. The instrument will be calibrated at least once daily and every 10 samples and results recorded on the Field Meter Instrument Calibration Log. Field replicates will be done at a frequency of every 10 samples. This will be done in quadruplicate. All field sample measurements will be recorded on the Well Purging and Sample Collection form.

15. Quality Control Requirements.

Quadruplicate samples will be done every 10 samples by rinsing the electrode after the initial reading, waiting 1 full minute, and then measuring the next sample. The standard deviation of four replicate measurements must be less than or equal to 0.1 standard unit.

16. References.

Material for this SOP was obtained from the Instruction Manual for the Fisher Accumet Model 955 Portable pH/mV Temperature Meter, Catalog No. 69348.

17. Method Validation Data.

The parameter of pH is being measured for field screening to select sampling locations and method validation data is, therefore, not required.

ARCS/P/HIMCO/AF2

Standard Operating Procedure
for the
Measurement of Specific Conductance in the Field
Using the Cole Parmer Model 4070 Conductivity Meter

1. Parameter to be measured: Specific Conductance.

2. Range of Measurement: Range (a) 0 to 19.99 mS
 (b) 0 to 1999 uS
 (c) 0 to 199.9 uS
 (d) -30.0 to +150.0°C

Resolution (a) 0.01 mS
 (b) 1 uS
 (c) 0.1 uS
 (d) 0.1°C

3. Sample Matrix.

This method is applicable to drinking, surface, saline, and groundwater samples; domestic and industrial wastes.

4. Principle, Scope, and Application.

The specific conductance of a sample is measured using a self-contained portable conductivity meter equipped with an Automatic Temperature Compensation (ATC) probe.

5. Interferences and Corrective Action.

- 5.1 Precipitation reactions and absorption of carbon dioxide from the air can affect the specific conductance. This can be avoided by taking the measurement as soon after sampling as possible.
- 5.2 Specific conductance is affected by temperature changes. The use of the ATC probe will eliminate this problem.
- 5.3 Any constituent of the sample which can coat the conductivity probe such as oil or grease, algae, or sedimentation can cause incorrect or sluggish response. Dried salts or particulate matter allowed to build up on the cell plates may cause the readings to be erroneous. It is important to rinse the cell carefully with deionized water.
- 5.4 Ensure that no air bubbles are trapped in the cell area between the plates. Failing to do so could cause a false reading.

6. Safety Precautions.

Common sense is the best safeguard against any potential hazards. Obviously, one must never pipette samples or standard solutions by mouth. Handle the probes carefully to avoid breakage. Since the meter will be measuring potentially hazardous material, the probes must never be placed in the mouth.

7. Sample Size, Collection, Preservation, and Handling.

Samples should be collected in clean glass or plastic containers. The volume should be such that the cell plates of the probe are completely immersed in the sample. Samples may be filtered through a 4.5 μ filter before analysis if necessary. Specific conductance should be measured as soon after sampling as possible, however, if readings cannot be taken immediately, samples should be stored at 4°C and measured as soon as possible.

8. Apparatus and Material.

- 8.1 Cole Parmer Conductivity Meter, Model 4070
- 8.2 Conductivity probe
- 8.3 Automatic Temperature Compensation (ATC) probe
- 8.4 Clean sample containers
- 8.5 Squeeze-type wash bottle, 125 ml or larger
- 8.6 Deionized water
- 8.7 Calibration standard solution
- 8.8 Laboratory wipes - KimWipe or equivalent
- 8.9 Replacement batteries, types PP3, 6F12, or MN1640

9. Routine Preventative Maintenance.

It is necessary to keep the external surfaces clean and free from dust.

All conductivity probes should be thoroughly rinsed after use and stored in deionized water. Particular care should be taken to ensure that the electrical connections are free from dirt and debris. It is recommended that the instrument be checked and calibrated before going into the field. The symbol "BAT" will appear on the display if the batteries need replacing.

10. Reagents and Calibration Standards.

Potassium chloride standard solutions with a specific conductance closest to the values expected in the field should be used. The table below may be used for guidance:

SPECIFIC CONDUCTANCE OF KCl SOLUTIONS AT 25 DEGREES CENTIGRADE

<u>Concentration</u> mol/l	<u>Specific Conductance</u>	
	mg/l	umhos/cm
0.0001	7.456	14.94
0.0005	37.28	73.90
0.001	74.56	147.0
0.005	372.8	717.8
0.00702	523.4	1000
0.01	745.6	1413
0.02	1591.2	2767

NOTE: This table has been modified from Standard Methods for the Examination of Water and Wastewater (1980).

Standard solutions may be stored at 4°C for up to one week. Date of preparation can be noted on the container and on the Field Meter Instrument Calibration Log.

11. Calibration Procedures.

Cole Parmer Model 4070 Conductivity Meter

Two options for calibration of the Model 4070 are available in the field, depending on instrument accessory availability. If the pre-calibrated probe is available, this is the easiest calibration procedure to do in the field. However, if the probe is not available, calibration will have to be done daily before field use with a standard solution (commercially prepared):

Calibration with Pre-Calibrated Probe:

- a. Connect a standard pre-calibrated probe to the unit.
- b. Select the "Set K" position on the function switch.
- c. Adjust the "Set K" control until the display indicates the value of the cell constant as marked on the probe being used.
- d. The standard X 1.0 probe has a cell constant range from 0.80 to 1.20 and is calibrated to 2 decimal places. The display should be set to indicate this figure exactly.

The standard X 10 probe has a cell constant range from 8.0 to 12.0 and is calibrated to 1 decimal place. The display should be set to indicate this figure shifted one place to the right.

The standard X 0.1 probe has a cell constant range from 0.08 to 0.12 and is calibrated to 2 decimal places. The display should be set to indicate this figure shifted one place to the left.

- d. Before using the 20 uS range, the probe should be thoroughly rinsed in deionized water, excess water shaken off and the outside of the probe body wiped dry. The display should then be set to zero, with the probe in free air, by using the "Zero 20 uS" control.

Calibration with a Standard Solution:

- a. Select the "COND" range.
- b. Immerse the conductivity cell and A.T.C. probe (if separate probe is being used) into the prepared standard.
- c. Select the 2000 uS range if using a 1000 uS or 1413 uS standard or other suitable range if an alternative standard is being used.
- d. Adjust the "Set K" control to give the exact readout of the standard solution selected on the display.
- e. After carrying out the calibration with standard solutions as detailed above and with the probe still in the standard solution and the display set to the value of the standard solution, switch to the "Set K" range to give a direct readout of the cell constant.

- f. If the readout cannot be set to the value of the standard solution with the "Set K" control, then the cell constant lies outside the "Set K" range of 0.80 to 1.20. In such cases, the display should be set to read 1.00 on the "Set K" position, the display reading on the range giving the best resolution should be noted and the cell constant calculated from the following formula:

$$K = \frac{\text{Conductivity of Standard Solution}}{\text{Display Reading}}$$

This figure should be noted and (with the cell constant set to 1.00 on the "Set K" position) the display reading should be multiplied by this figure to obtain the correct conductivity.

Note:

Ensure that the conductivity standard has not reached or gone over the expiration date marked on the container.

12. Sample Preparation.

If the sample contains a great deal of particulate matter which may interfere with readings, they may be filtered through a 4.5 m filter.

13. Analytical Measurement.

- 13.1 Calibrate the instrument (See Section 11).
- 13.2 Rinse the conductivity probe and the ATC probe thoroughly with deionized water, shake to remove internal droplets, and the outside should be wiped before immersing into sample.
- 13.3 Allow the readout on the instrument to settle (usually about one minute). Multiply the instrument readout by the correction factor determined during calibration. Record the corrected conductivity on the Well Purging and Sample Collection form.
- 13.4 Step 13.2 should be repeated after every sample to prevent cross-contamination.
- 13.5 On completion of sample measurement, the probes should be thoroughly rinsed in deionized water and for short term storage should be kept immersed in deionized water so that the plates remain wetted. For longer term storage, the probes should be thoroughly rinsed in deionized water, the outside of the probes wiped dry, and the probes stored dry. It should be noted that it may take some time for stability to be achieved when a dry probe is first used while the plate becomes re-wetted.

14. Data Deliverables.

Initial calibration of the instrument and continuing calibration check results will be recorded on the Field Meter Instrument Calibration Log form. Summary of sample analysis will be recorded on the Well Purging and Sample Collection form, including sample duplicates.

15. Quality Control Requirements.

The meter will be read to the nearest 10 umhos/cm within a range of 0 to 20,000 umhos/cm. Accuracy of measurements shall be ± 5 percent of a standard. The meter will be calibrated at least once daily and after every 10 field samples. Field duplicates will be measured at a frequency of every 10 samples by thoroughly rinsing the probes in deionized water, waiting one full minute, and then immersing probes in the duplicate sample. Precision shall be a standard deviation of ± 10 percent.

16. References.

Material for this SOP was obtained from the Instruction Manual for the Cole Parmer Model 4070 Conductivity Meter.

17. Method Validation Data.

The parameter of specific conductance is being measured for field screening to select sampling locations, and method validation data is, therefore, not required.

ARCS/P/HIMCO/AF4

Standard Operating Procedure for Field Measurement of Dissolved Oxygen

I. PARAMETER

Dissolved Oxygen in mg/l

II. RANGE OF MEASUREMENT

0-10 and 0-20 mg/l

0-5 and 0-10 mg/l with high sensitivity membrane

III. LIMIT OF DETECTION

0.05 mg/l on 0-10 scale

0.1 mg/l on 0-20 scale

IV. SAMPLE MATRIX

Water (surface, groundwater), residential well, wastewater

V. PRINCIPLE SCOPE AND APPLICATION

A thin permeable membrane covers a polarographic sensor. Oxygen enters the membrane, and when a polarizing voltage is applied across the sensor, oxygen reacts at the cathode causing a current to flow. The membrane passes oxygen at a rate proportional to the pressure difference across it. Oxygen is consumed at the cathode so the oxygen pressure inside the membrane is zero. The force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

VI. INTERFERENCES AND CORRECTIVE ACTIONS

Air bubbles trapped under the membrane will cause pressure-induced errors. Replace membrane after refilling with fresh electrolyte taking care not to introduce air bubbles.

H₂S, SO₂, halogens, neon, nitrous oxide, and CO are interfering gases. For water samples with chloride concentrations of greater than 20,000 mg/l, compensation must be made for the differing temperature solubility relationship of oxygen in sea water.

If the gold cathode becomes tarnished from contact with certain gases or plated with silver from extended use with a loose or wrinkled membrane, it will need to be replaced. Do not attempt to clean the cathode.

VII. SAFETY PRECAUTIONS

Disconnect battery charger before removing cover to replace batteries. When replacing batteries, positive end of battery must go to red.

VIII. SAMPLE SIZE, COLLECTION, PRESERVATION, AND HANDLING

Samples should be collected in 200 ml BOD bottles directly from the surface water body or residential tap with minimum agitation to entrain air. No preservatives are applicable as measurement should be done immediately after collection. Downhole measurement of dissolved oxygen in monitoring wells should be done.

IX. APPARATUS AND MATERIALS

YSI Model 54ARC/54ABP dissolved oxygen meter
YSI 5720A self-stirring BOD bottle probe
YSI 5739 oxygen temperature probe
Battery charger eliminator
Detachable cable leads (10-200' as applicable)
Submersible stirrer
Calibration chamber
Standard membrane and KCl Kit (includes membrane packets (0.001" thick), 30 ml KCl bottle with Kodak Photo Flo®)
High sensitivity membrane and KCl Kit (includes membrane packets 0.0005 " thick)
Spare O rings
BOD bottles
Scissors
Spare Ni-Cad 1.25 v batteries

X. ROUTINE PREVENTIVE MAINTENANCE

Check gold cathode for tarnish or plating monthly. Send in for service annually.

Check probe for air bubbles and membrane for fouling daily and replace as necessary. Average life is 2 to 4 weeks.

Check diaphragm for puncture and evidence of leakage of electrolyte weekly. Replace, if needed, by unscrewing the retaining plug. Remove diaphragm and washer, and flush any KCl crystals from the reservoir. Install new diaphragm, convoluted side in, replace washer, and screw in retaining plug.

Rinse stirrer after each sample to maximize its life as hydrocarbons, ozone, and strong acid and bases may degrade the flexible stirrer boot.

Store probe in the bottomless plastic bottle with a small piece of moistened towel or sponge to keep electrolyte from drying out. Alternatively, store probe in BOD bottle with about one-inch of water.

Battery replacement or recharging is indicated if the red line adjustment cannot be made or calibration cannot be achieved even after probe replacement/maintenance. Battery life should be three years or longer. Recharge a minimum of 16 hours with the instrument on.

XI. REAGENTS AND CALIBRATION STANDARDS

Electrolyte solution available through YSI or make as follows:

1. Prepare a saturated solution of KCl using reagent grade KCl and distilled water.
2. Dilute saturated solution to half strength using distilled water.
3. Add two drops Kodak Photo Flo® per 100 ml solution to provide good wetting of the sensor.
4. Store with meter, discard after one year.

XII. CALIBRATION PROCEDURES

The air calibration procedure described below should be done daily before field use:

1. Place the probe in the YSI 5075A calibration chamber along with a few drops of distilled water.
2. Wait approximately 10 minutes for temperature stabilization.
3. Switch to TEMPERATURE and read. Refer to Table 1 - Solubility of Oxygen in Fresh Water and determine calibration value.
4. Determine altitude or atmospheric correction factor using Table II.
5. Multiply Table I calibration value by the Table II correction factor.
6. Switch to the appropriate mg/L range and adjust the CALIBRATE knob until the meter reads the value calculated in Step 5. Wait two minutes to verify calibration stability. Readjust if necessary.

Table 1 shows the amount of oxygen in mg/l that is dissolved in air saturated fresh water at sea level (760 mmHg atmospheric pressure) as temperature varies from 0° to 45°C.

TABLE 1
SOLUBILITY OF OXYGEN IN FRESH WATER

<u>Temperature</u> <u>°C</u>	<u>mg/l Dissolved</u> <u>Oxygen</u>	<u>Temperature</u> <u>°C</u>	<u>mg/l Dissolved</u> <u>Oxygen</u>
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derived from 15th Edition "Standard Methods for the Examination of Water and Wastewater."

Table II shows the correction factor that should be used to correct the calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmospheric pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

TABLE II
CORRECTION FOR ATMOSPHERIC PRESSURE

Atmospheric Pressure <u>mmHg</u>	or	Equivalent Altitude <u>Ft.</u>	-	Correction <u>Factor</u>
775		540		1.02
760		0		1.00
745		542		.98
730		1094		.96
714		1688		.94
699		2274		.92
684		2864		.90
669		3466		.88
654		4082		.86
638		4756		.84
623		5403		.82
608		6065		.80
593		6744		.78
578		7440		.76
562		8204		.74
547		8939		.72
532		9694		.70
517		10472		.68
502		11273		.66

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

XIII. SAMPLE PREPARATION

None required, analyze immediately.

XIV. ANALYTICAL MEASUREMENT

1. With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and provide stirring by:
 - a. Downhole well measurement - use YSI submersible stirrer, or provide manual stirring by raising and lowering the probe about 1 ft/sec.
 - b. Surface water and tap water measurements - place sample in BOD bottle and use self-stirring BOD bottle probe or magnetic stirrer.
2. Allow sufficient time (at least one minute) for probe to stabilize to sample temperature and dissolved oxygen.
3. Read dissolved oxygen and record on Well Purging and Sample Collection Form.

XV. FLOW CHART

Not applicable.

XVI. DATA TREATMENT

Not applicable, read dissolved oxygen directly in mg/l.

XVII. DATA DELIVERABLES

Complete Field Meter Instrument Calibration Log and note any preventive maintenance done. Record sample dissolved oxygen, date, time, and analyst on Well Purging and Sample Collection Form under comments column.

XVIII. QUALITY CONTROL REQUIREMENTS

No blanks, spikes, reference, or QC samples are applicable.

Every tenth sample analyzed, or at least once daily, sample will be analyzed in duplicate by collecting a fresh aliquot of sample or raising the submersible probe and reintroducing it into the well to take a duplicate dissolved oxygen measurement. Error associated with just the instrument is ± 0.05 mg/l or 5%, therefore, field duplicates are likely to agree within ± 0.1 mg/l or 10%. If the variation is larger, check instrument probe, battery, and diaphragm conditions.

XIX. REFERENCES

1. Instruction Manual YSI Models 54ARC/54ABP Dissolved Oxygen Meters, YSI Incorporated, Yellow Springs, Ohio, August, 1988.
2. YSI Product Service (513) 767-7241 or 1-800-343-HELP.

XX. METHOD VALIDATION DATA

Not required, dissolved oxygen measurement used as field screening only.

A/O/MISC/BB7

Standard Operating Procedure for the Field Measurement of Radioactivity

I. PARAMETERS

Total alpha radiation
Total beta radiation
Total gamma radiation

II. RANGE OF MEASUREMENT

At x 1 range rocker switch: 0-5 mR/h
At x 100 range rocker switch: 0-500 mR/h
or with external probe:
At x 1 range rocker switch: 0-5,000 cpm
At x 100 range rocker switch: 0-500,000 cpm

III. LIMIT OF DETECTION

1 mR/h or 1 cpm at X 1 range
100 mR/h or 100 cpm at x 100 range

IV. SAMPLE MATRIX

Soil collected during the installation of monitoring wells and the land-fill cap investigation will be analyzed.

V. PRINCIPLE, SCOPE, AND APPLICATION

A halogen quenched Geiger - Mueller tube is used as the detector in the Mini CONRAD II. The Mini CONRAD II is intended to be used as a survey instrument by itself and a contamination meter with the external probe. In order to quantitatively assess the radioactivity present in site soils, the external probe accessory will be used to measure total alpha, beta, and gamma contamination in counts per minute (cpm).

VI. INTERFERENCES AND CORRECTIVE ACTION

The operating temperature range is from -12°F to +122°F. Extremely cold weather may result in erroneous readings. Use in a warmer environment and bring sample to the meter rather than using it directly at the sample collection site.

If the speaker or 9-volt battery is oriented so that it is between the detector (probe) and sample to be measured or calibration source, the instrument will not respond with the stated accuracy of 10%. Ensure that the GM tube indicator target mark on the probe is aimed toward the sample or calibration source.

VII. SAFETY PRECAUTIONS

The overflow circuitry insures that the meter will continue to read full-scale in fields up to 100 times the maximum range, giving an indication of high radiation levels so the user can avoid exposure. However, the user should exercise caution in working in any suspected radioactive environment.

The instrument has been drop-tested at heights of up to four feet onto concrete. Caution should still be used in protecting the geiger tube (probe) assembly.

VIII. SAMPLE SIZE, COLLECTION, PRESERVATION, AND HANDLING

Samples will be analyzed in the field directly from the split spoon or sample trowel. No preservation is required.

IX. APPARATUS

Mini CONRAD II Portable Radiological Survey Meter
Meter 3089 Probe

X. ROUTINE PREVENTIVE MAINTENANCE

Semi-annual calibration by the manufacturer, Dosimeter Corporation, is recommended. When the instrument is stored for longer than one week, the 9-volt battery should be removed.

XI. REAGENTS AND CALIBRATION STANDARDS

The target center calibration standards is supplied with the instrument. No shelf life or storage is applicable.

XII. CALIBRATION PROCEDURES

The manufacturer recommends that the Mini CONRAD only be calibrated semi-annually. Determine from the instrument log the last date of calibration and assess if any in-field calibration will be needed. The calibration procedure is as follows:

1. Place the range switch in the low (x1) position.
2. Remove the "x1" plastic screw that covers the low-range calibration control.
3. Turn the Mini CONRAD II on and place the target center in the appropriate field for the instrument being calibrated: Model 3034 3 mR/h.
4. Adjust the "x1" control for the proper indication on the meter scale.
5. Reduce the field by approximately 50 percent. The Mini CONRAD II must indicate with 15 percent of the field value.

6. Replace the "x1" plastic screw.
7. Place the range switch in the high (x100) position, remove the "x100" plastic screw and place the Mini CONRAD II target center in the appropriate field: Model 3034 300 mR/h.
8. Adjust the "x100" control for the proper indication on the meter scale. The field intensity is 100 times the value indicated.
9. Reduce the field by approximately 50 percent. The Mini CONRAD II reading, multiplied by 100, must be within 15 percent of the field.
10. Replace the "x100" plastic screw and turn the Mini CONRAD II Off.

Note:

For calibration purposes, the "target center" is the intersection of the targets on the case.

XIII. SAMPLE PREPARATION

No sample preparation is necessary, probe is to be pointed at sample contained in the split spoon or trowel.

XIV. ANALYTICAL MEASUREMENT

1. Check the battery by putting the power switch in the BATT (left) position. The switch must be held in this position since it is spring-loaded and will return to the OFF position when released.
2. The meter should indicate a reading in the BATT range. If the reading is below, the battery should be replaced as less than 8 hours of battery life remain.
3. Press the power switch to ON (right) position.
4. Plug in the probe and point the probe at the sample (within 2-3 inches).
5. Select the desired range x1 or x100 with the range switch. Read the cpm scale. For most sites/samples, the x1 range is most applicable.
6. If the x100 range is used, be sure to multiply the cpm reading by 100.
7. Record the cpm on the Soils Data Form.

XV. FLOW CHART

Not applicable.

XVI. DATA TREATMENT

Instrument is direct readout unless x100 range is used. If x100 range is used, multiply readings by 100.

XVII. DATA DELIVERABLES

Not applicable. Record reading on Soils Data Form.

XVIII. QUALITY CONTROL REQUIREMENTS

One soil sample for every ten measured should be split into two aliquots and have a reading taken on each. Both measurements should be recorded on the Soils Data Form, and the rpd calculated. It is expected that readings will agree to within 15%.

An off-site background soil will be measured daily to determine background counts.

XIV. REFERENCES

Instruction Manual, Mini CONRAD II Portable Survey Meter

Dosimeter Corporation (513) 489-8100

XX. METHOD VALIDATION DATA

Radioactivity is being measured for field screening and health and safety purposes, so method validation data is not required.

A/O/MISC/BB9

Standard Operating Procedure for the
Field Measurement of Methane and Hydrogen Sulfide

I. PARAMETERS

Methane (CH_4), as % LEL
Hydrogen sulfide (H_2S), as ppm

II. RANGE OF MEASUREMENT

CH_4 0-100% LEL
 H_2S 0-100 ppm

III. LIMIT OF DETECTION

CH_4 1% LEL
 H_2S 1 ppm

IV. SAMPLE MATRIX

Gases, vapors, ambient air.

V. PRINCIPLE, SCOPE, AND APPLICATION

A catalytic sensor provides a linear output to the meter circuit to read the presence of combustible gas. As the instrument is factory-calibrated to methane, the reading is equated with % LEL as methane. An electrochemical reaction sensor provides measurement of H_2S .

VI. INTERFERENCES AND CORRECTIVE ACTION

Water drawn through the sampling pump will contaminate the pump and sensors. Attach a water trap and filter device as a preventive measure. If water is sucked into the pump, open the instrument and dry it quickly with a warm air drier as soon as possible. The unit must then be sent in for service.

VII. SAFETY PRECAUTIONS

The Gasponder IV is designed to be intrinsically safe for operation in confined spaces with potentially hazardous atmosphere. However, no instrument can completely guarantee that the atmosphere is free of contamination simply because no readings are obtained.

The instrument itself should only be charged with its own AC charger. No substitutions should be made. The calibration gas cylinders should be handled carefully in accordance with good laboratory practices.

VIII. SAMPLE SIZE, COLLECTION, PRESERVATION, AND HANDLING

The Gasponder IV sample intake tubing will be placed in the soil gas probe or near suspected entry locations for CH₄/H₂S in residential basements, and the CH₄ and H₂S readings taken immediately.

IX. APPARATUS AND MATERIALS

Gasponder IV PGM-14
Tygon tubing
Battery pack and spare
Thermal-protected charger
Water trap and filter device
Methane/CO calibration cylinder
H₂S calibration cylinder
Gas regulator and hose

X. ROUTINE PREVENTIVE MAINTENANCE

Only trained and authorized Lumidor personnel should attempt any maintenance or repairs other than calibration and replacement of the battery pack.

Calibration is discussed in Section 12. Battery pack replacement should be done when the digital meter face indicates LO-BAT.

XI. REAGENTS AND CALIBRATION STANDARDS

Two calibration gas cylinders are supplied with the instrument:

CH₄/CO (50% LEL and 250 ppm CO)
H₂S (25 ppm)

New cylinders should be reordered from Lumidor when the regulator indicates that the cylinder is near empty.

XII. CALIBRATION PROCEDURES

1. Initial start-up and zero set

- a. Place selector at any "ON" position. All alarm lights should be "ON." Alarm horn should activate for several seconds, and display will indicate 1.888.
- b. Turn selector switch to "BATT. TEST." Reading should be 4.9V minimum.
- c. If less than 4.9V, place on charge for several hours.
- d. Attach sampling hose and pinch tightly. "LOW FLOW" alarm should indicate and horn should sound.

- e. Instrument should be run at least 15 minutes.
- f. Place selector on "BATT. TEST" - check reading.
- g. Place selector on H₂S and set "ZERO."

2. Calibration

- a. Locate opening provided at side of main case to gain access to calibration adjustment for H₂S and CH₄.
- b. Attach flow regulator on Methane/CO cylinder.
- c. With selector set to "CH₄," check "ZERO" setting. If necessary, readjust with panel adjustment only.
- d. Repeat step 2 for "H₂S."
- e. Return selector switch to the "CH₄" position. Turn on flow regulator, and connect hose to unit using quick disconnect fitting.
- f. After one minute, adjust calibration screw until reading is the same as stated on cylinder label. (i.e. 50% LEL).
- g. Turn off flow regulator and remove regulator from cylinder.
- h. Attach flow regulator to cylinder marked H₂S (RED).
- i. Turn selector switch to the H₂S position. Turn on flow regulator and connect hose to unit using quick disconnect fitting. After three minutes, adjust calibration screw until reading is the same as stated on cylinder (i.e. 225 ppm). Disregard "O₂" alarm that occurs during this test.
- j. When calibration procedure is completed, remove the gas regulator from cylinder, carefully repack all components, and replace calibration cover securely.

XIII. SAMPLE PREPARATION

Not applicable, analysis is done directly on waste mass gas in sampling probe or residential air.

XIV. ANALYTICAL MEASUREMENT

- 1. Attach sampling hose to gas inlet and lower into probe. Do not allow hose to contact any water or leachate. In residential basements, place hose near cracks in foundation or sump pump areas.
- 2. Switch dial to "Read ppm H₂S." Record on Soils Data Form.

3. Switch dial to "Read & LEL Methane." Record on Soils Data Form.

4. Remove hose from probe or sampling area, and proceed to next sampling location.

XV. FLOW CHART

Not applicable.

XVI. DATA TREATMENT

Not applicable. Record results of calibration on Field Meter Instrument Calibration Log and results on Soils Data Form.

XVIII. QUALITY CONTROL

Not applicable as measurements are real time direct read-outs. An upwind off-site location will be measured daily before field work to determine background conditions.

IX. REFERENCES

1. Lumidor Safety Products Operating Instructions for Gasponder IV
Model PGM-14, May, 1987, Revision I.

2. Lumidor Product Service (305) 625-6511.

XX. METHOD VALIDATION DATA

Not applicable as CH₄ and H₂S are being measured for field screening to select sampling locations.

A/O/MISC/BB7